PROCESS DESIGN FOR IGUN ORE DEPOSIT IN ILESHA GOLDFIELD

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DECLARATION

I hereby declare that this thesis was composed by me and it is a record of my research work. It has not been accepted in any previous application for a higher degree. All sources of information are specifically acknowledged by means of refrences.



JOHN ADE AJAYI

02/03/98

DATE

CERTIFICATION

This thesis entitled **Process Design For Igun Ore Deposit in Ilesha Goldfield** by **Engr. John Ade Ajayi** meets the Regulations governing the award of the degree of **Doctor of Philosophy** of Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

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DEDICATION

This thesis on the king of metals is dedicated to my beloved wife, **'Laide**, who is far more precious than gold; our son, **Iseunifẹ**, a good and perfect gift from God; **late Dr. G.G.O.O. Uwadiale**, a doyen of mineral processing; other **painstaking and honest researchers in the mineral industry**; and to **all who are preparing for the imminent return of the King of kings, our Lord Jesus Christ** for here we have no continuing city but we seek one (the golden city of the heavenly Jerusalem) to come.

QUOTABLE QUOTES

"God hath spoken once; twice have I heard this; that power belongeth unto our God" Psalm 62:11

"The roots of education are bitter, but the fruit is sweet" - Aristotle.

If I have seen further, it is because I have stood on the shoulders of giants" - Isaac Newton.

" I shall be telling this with a sigh,
Somewhere ages and ages hence:
Two roads diverged in a wood,
And I took the one less travelled by,
And that has made all the difference " - Robert Frost in That Road Not Taken.

"The greatest thing in the world is not so much where we are, but in what directions we are moving" - Oliver Wendel Holmes.

"If we could first know where we are and wither we are tending, we could better judge what to do and how to do it " - Abraham Lincoln.

"Those things that hurt instruct" - Benjamin Franklin.

"It is always darkest just before the day dawneth" - Thomas Fuller.

"We triumph without glory when we conquer without dangers" - William Davies.

"We are the circumcision which worship God in the spirit, and rejoice in Christ Jesus, and have no confidence in the flesh " Philipians 3:3

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ABSTRACT

Occurrence of economic quantities of gold deposits over a large expanse of area and in finely disseminated form has been reported in Ilesha (Western Nigeria). The problem, however, has been inavailabity of appropriate technology for efficient recovery of gold. This is the thrust of this work.

This investigation, process design for Igun ore deposit in Ilesha goldfield, is aimed at proposing process flowchart for efficient recovery / extraction of gold from Ilesha gold ore accentuating on Igun deposit. The research essentially entails preliminary investigation, mineral processing and mineralogical characterisation of the ore which involve sampling, concentration, particle size analysis, ore chemical analysis by fire-assay-gravimetric and atomic absorption spectrophotometric methods, petrography and x-ray diffractometry. In addition, amenability assessment of Igun gold deposit to amalgamation and percolation leaching was carried out.

It has been established that free gold is scanty in the ore, rather the gold occurs as specks and inclusions finely disseminated in sulphide and quartz phases. It is postulated that Ilesha alluvial gold ore of which Igun gold deposit is a paradigm belongs to a non-refactory class. The experimental results confirm this as the gold ore is amenable to percolation cyanidation leaching but responded poorly to amalgamation. At the optimum leaching parameters of 6-hour retention time, sodium cyanide concentration of 0.3% and hydrogen ion concentration (pH) of 11.5, the highest recovery (63.60%) was obtained with the smallest size fraction of -75μm. These results form the basis for the evolution of a process flowchart for recovery of gold from Igun ore deposit which is aimed at sustainable gold production under safe environmental conditions.

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CHAPTER ONE

INTRODUCTION

1.1 History of Gold Mining in Nigeria

Areas constituting what may be described as the gold belt of Nigeria are Zamfara-Malenjo area in Sokoto State, Lapai-Beni-Gwaderi area in Niger State, old Birinin Gwari in Kaduna State and Ilesha-Ife area of Osun State. Exploration work recently embarked upon by the Nigerian Mining Corporation in Niger State covers the vast expanse of land between Beni in South East and Gwaderi in the North West. Most mining activities in this area were carried out before independence. To date, the foundation of the old mining camps, pits and heaps of pebbles can still be seen. The result of the exploration work shows that very low gold values were obtained, thus the economic exploitation of the deposit may not be justified.

At Ilesha area gold mining had been extensive in the past by relatively simple labour intensive methods but this activity ceased about forty years ago. Preliminary investigations recently carried out by the Nigerian Mining Corporation indicate that gold is still present in good values over a wide area in both primary and alluvial deposits (Anon, 1981). In 1936, there was a sharp rise in the number of goldsmiths in Ijeshaland and this led to the suspection of gold occurrence in the area but it was not until September 1940 that the discovery of Ilesha goldfield was reported by Mr. Gloucester Trotman. Organised (Anon, 1981) mining started in December 1941 after pegging of a lease by Messrs Harper and Passman in August of the same year, and peak production was reached in 1942 at Ilesha goldfield. Records from the Western Inspectorate of Mines Department, Ministry of Solid Minerals Development showed that 22,065 ounces (486.39 kg) of raw gold was won in 1942, 80 ounces (2.49 kg) in 1941, 8,114 ounces (252.37 kg) in 1943 and 2,149

ounces (66.84 kg) in 1944. Since mining began in Nigeria in 1914, a total of 379,330 troy ounces (approximately 11,789.49 kg) of gold was produced up to 1985. The production from this area has contributed significantly to the national output of gold as illustrated in Figures 1.1 and 1.2.

1.2 Ilesha Gold Belt

Ilesha gold deposit lies within latitude $7^0 30'$ and $7^0 35'$ North and longitude $4^0 23'$ and $4^0 38'$ East covering a total area of about 23 square kilometres as shown in Figures 1.3 and 1.4. The area lies within the special exclusive prospecting licence No. 225 of Nigerian Mining Corporation and constitutes part of Ilesha/Ife gold field covering 200 square kilometres. The Mining rights are vested with Nigerian Mining Corporation for whom additional lease was pegged during the project (ML 20501). This adjoins mining lease (ML 10904) earlier pegged by Metals and Minerals for Nigerian Mining Corporation.

1.3 Problems Associated with Ilesha Gold Exploitation

The life of mining at Ilesha gold field was short since no prospecting was used to support the mining and everything came to an abrupt end with the exhaustion of the 'proven' areas. The mining methods employed were primitive and wasteful. None of the operators had any mining training and their experience was what they acquired with the assistance from Government officials. The practice started with haphazard digging and panning. Following this, wooden sluice boxes were introduced and towards the end of the first mining lease of the area, organised and small paddocks, pumpsunks and few drainage ditches were employed. This practice which was far from good mining left substantial amount of the mineral in the earth.

Furthermore, processing of gold was such that substantial part of the gold values had to be discarded with the tailings (since the gold was said to be finely disseminated) because there was no economic means of processing

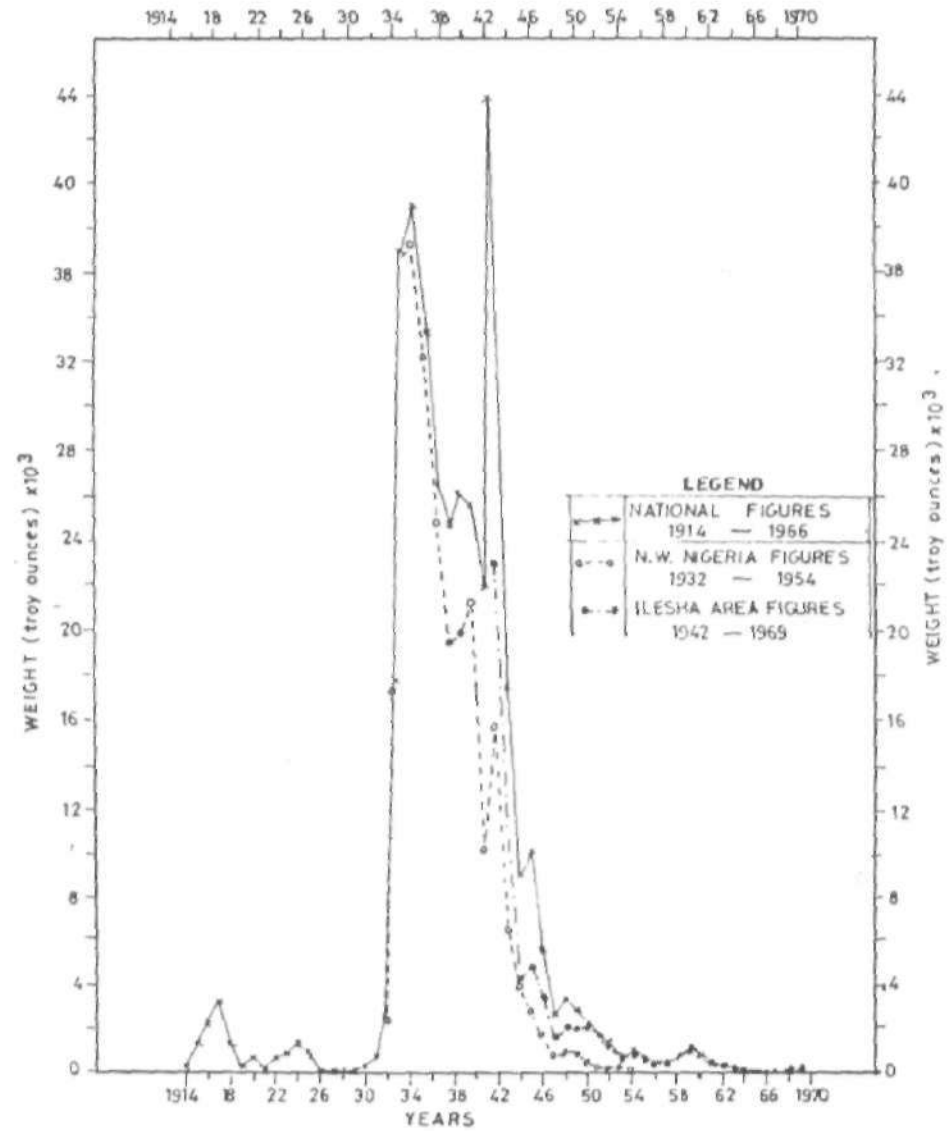


Fig.1.1 TOTAL ANNUAL GOLD PRODUCTION (1914-1969)
(SOURCE; FED. MIN. OF SOLID MINERALS DEVELOPMENT)

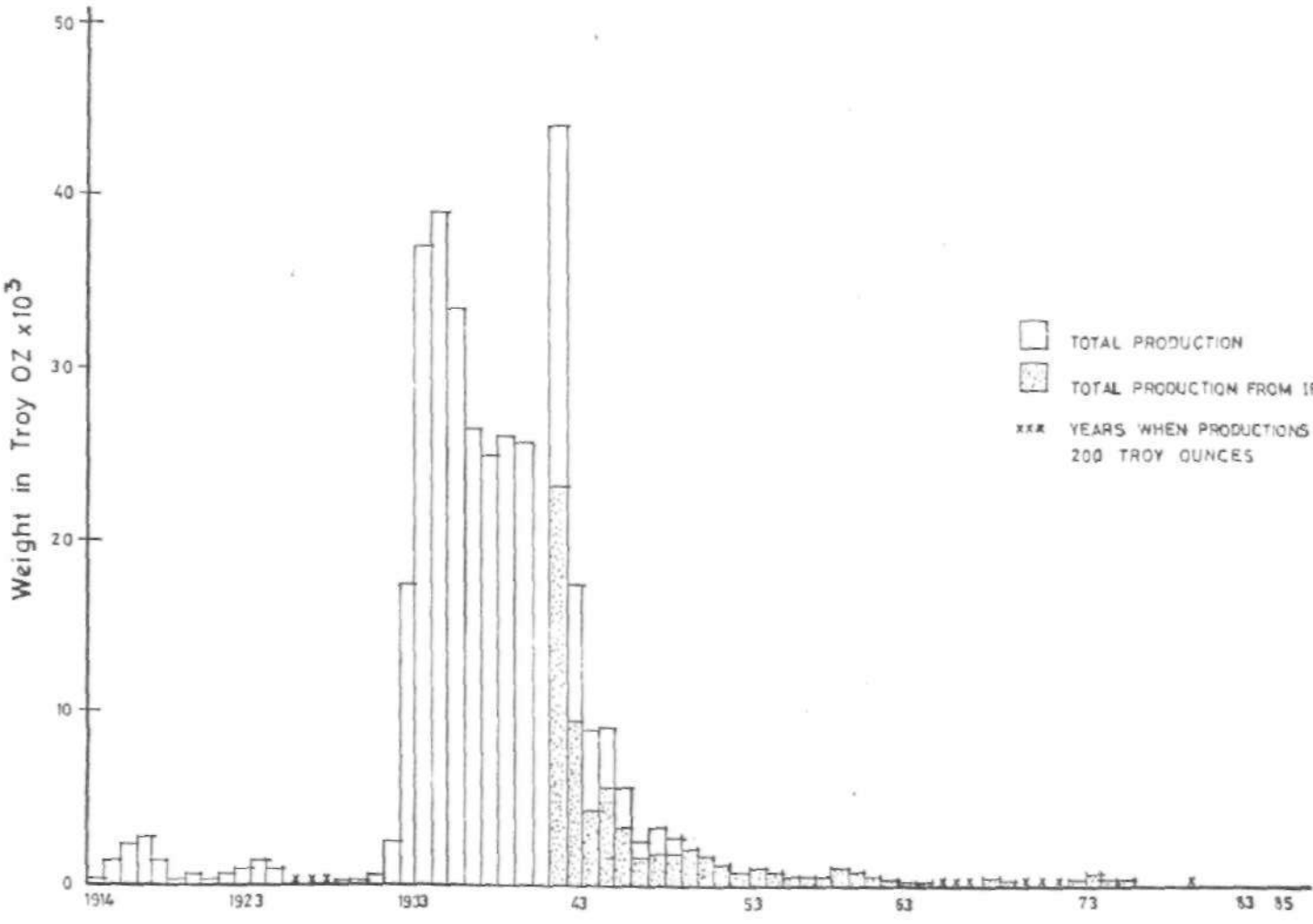


Fig. 1.2 Figure showing total annual gold production (1914 - 1985) from Nigerian Gold Fields compared with production from Ife-Ilesha area.
(SOURCE; FED. MIN. OF SOLID MINERALS DEVELOPMENT)

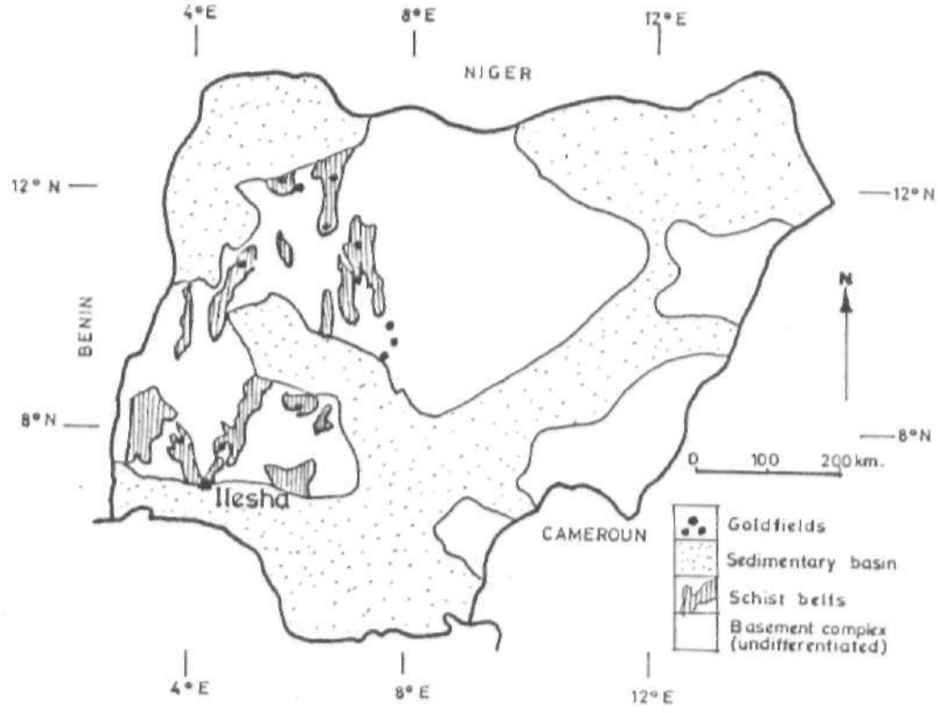


Fig. 1.3: Location map of Ilesha (S.W. Nigeria) Goldfield.
(SOURCE ; GARUBA I., 1988)

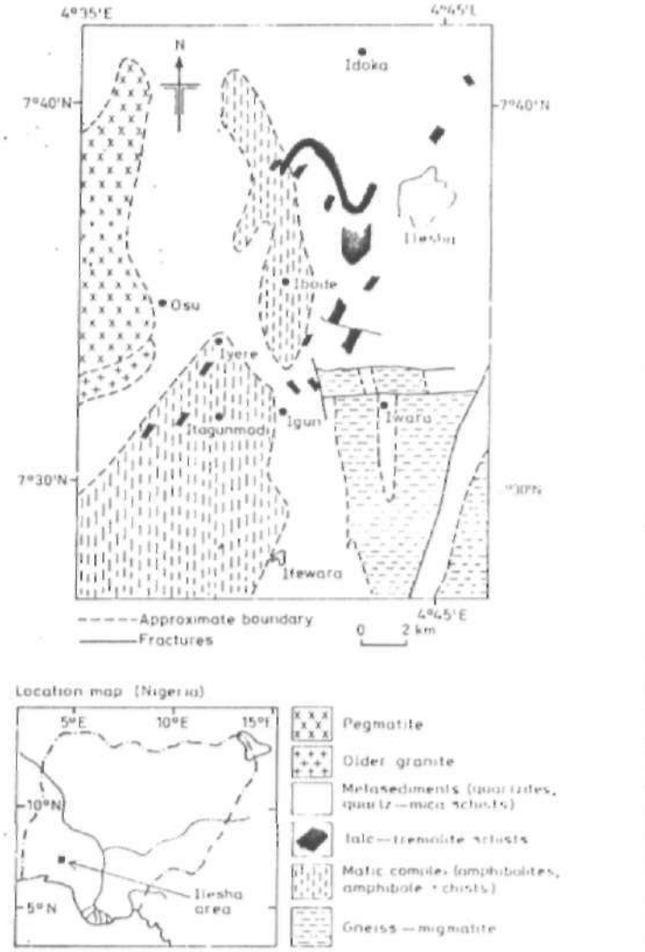


Fig 1.4 General geology and location map of Ilesha area
(After Elueze and Olade, 1985)

- by way of appropriate technology - that would effectively recover most of the purportedly fine gold in the ore. This is the main thrust of this study. It would require an indepth study of the problems associated with the processing and recovery of gold. The study therefore addresses the problems associated with processing and recovering of Ilesha gold.

1.4 Scope and Objectives of Investigation

In the Ife-Ilesha area two types of gold mineralization have been recognised:lode and placer deposits (De Swardt and Eric, 1953). Primary gold bearing quartz veins occur in feldspathatic gneiss in the amphibolitic complex around Itagunmodi, Ibodi, Igun, Iregun, Ilashe and surrounding areas. Records show that production from primary quartz veins is less significant than that from the alluvial gold associated with the amphibolite complex (Anon, 1981).

Although Ilesha gold consists of Iperindo reef which is covered by ML 19706 - area awaiting underground mining -, Itagunmodi alluvial deposit covered by ML 10904 (about 855.7 hectares), and Igun alluvial deposit, ML 20501 (2,276.8 hectares), this investigation centres on the alluvial deposits with particular reference to Igun gold ore. This is because of large expanse of Igun alluvial deposit and the availabilty of mineral processing equipment on the site.

The objective of this investigation is to come up with report proposing process flowchart for the recovery/extraction of gold from Igun alluvial gold ore. It is expected that the findings would provide valuable information for future exploitation of Ilesha alluvial gold ore.

The research involves the following:

(i) Preliminary investigation which entails sampling and concentration of Igun gold ore, particle size analysis; and mineralogical investigation on the ore using petrography and x-ray diffraction techniques.

(ii) Samples assay and instrumental analysis of Igun gold ore using the atomic absorption spectrophotometer and fire-assay-gravimetric techniques.

(iii) Laboratory investigations: Amalgamation, percolation-leaching and optimization of leaching parameters.

(iv) Proposal of process flowchart for Ilesha alluvial gold ore with Igun ore deposit as paradigm.

CHAPTER TWO

REVIEW OF LITERATURE

2.1 Geology and Mineralogy of Gold

The metallurgy of gold is shown by Holloway (1994) to be bound up with its geology and mineralogy, and an understanding of its metallurgy can only be achieved by understanding the way in which gold is found in the earth. The nature and types of gold occurrence have to be properly understood before the appropriate mineral processing technology could be devised. Petrographic and mineralogical studies of ores are important in the recovery study in that:

(i) the mineral constituents are identified,

(ii) the proportion in which they occur is established, and

(iii) the degree of dissemination of the valuable is ascertained.

2.1.1 Types of Gold Occurrence

There are two main types of gold occurrence: primary (consolidated or lode) deposits and placer (unconsolidated) deposits.

Primary Deposits

Ultimately, all gold originated from volcanic activity. Where found in undisturbed reefs or lodes, the deposit is said to be primary deposit and is frequently associated with sulphide minerals such as pyrite, arsenopyrite and stibnite.

There are three major groups of primary gold ores:

(i) Gold bearing quartz reefs: This type of reef contains only small amounts of sulphide minerals (0.5 - 3%), but in some places contains considerable

amount of scheelite ($CaWo_4$), Wolframite ($Fe,MnWo_4$)) and "mineralized" altered wall rock. Gold is visible at least under the microscope, and a large amount is liberated after coarse grinding to 0.5 - 0.8mm. Only 0.2 g/ton are enclosed in quartz or sulphide grains and cannot be recovered by gravity separation, amalgamation or cyanide leaching. Conventional gravity methods or plate amalgamation recover about 70 - 80%

(ii) Sulphidic banded Iron Stone: This type of primary deposit consists of bands of quartz and other silicates with pyrite (FeS_2) and pyrrhotite ($Fe_{1-x}S$) which contains some gold. The sulphides are reactive to air and water, and the reefs near the surface decompose leaving the gold behind as "free" gold frequently in association with hematite.

(iii) Complex, strongly sulphidic (3-15% sulphide) ores: Here, gold occurs in particles visible under the microscope or in a form recognisable only with electron microscope. Some free gold is mostly present but most of the gold is associated with pyrite (FeS_2), pyrrhotite ($Fe_{1-x}S$), arsenopyrite (FeAsS), stibnite (Sb_2S_3), galena (PbS), sphalerite (ZnS), chalcopyrite ($CuFeS_2$) and in complex ore minerals, and/or tied to tellurides. As gangue, quartz carbonates, mica, hornblende, augites and graphite may be present. These are refractory ores that do not yield their gold content when treated by the standard gravity and cyanidation processes.

Problable causes of refractoriness in gold ores include the following:

(1) Physical lock-up: Extremely fine gold particles encampsulated and therefore remain inaccessible to cyanide solution.

(2) Coated gold: When particles of gold closely associated with sulphides are in contact with water or lixiviant, they can develop coatings that

inhibit their dissolution in cyanide solution.

(3) Decomposition of host minerals: The sulphide can decompose to form cyanates, which combine with the available cyanide and thus deplete the 'free' cyanide that is required to dissolve the gold. Besides, oxygen is consumed so rapidly during decomposition of the mineral, that the gold-dissolution process which also requires oxygen is inhibited.

(4) Carbonaceous materials. The presence of carbon in an 'active' form will adsorb gold that has dissolved thus depleting the lixiviant.

(5) Coatings formed during rusting. Iron oxides, silicates and lead and antimony minerals can form coatings on gold particle during roasting.

(6) Insoluble alloys of gold. Although these are minor causes of refractoriness, the gold-silver alloy, gold-bearing tellurides and arsenides, and minerals such as aurostibnite ($AuSb_2$) and maldonite (Au_2Bi) have been found to be resistant to cyanidation. (Dry and Coetze, 1986; Nagy et.al., 1966; Sweigert and Leibenberg, 1966; Hansen and Laschinger, 1966; Hedley and Tabachnick, 1968).

The most common technique used for the recovery of gold from refractory ores is the roasting of flotation concentrates prior to cyanidation.

Placer Deposits.

Deposits formed by mechanical concentration where water, wind and other forces are involved in the process of rock breaking, transportation and deposition are called placer or detrital deposits. Placer can be eluvial, prolluvial and alluvial depending upon their position from the source and the major weathering agents. Placer deposits are said to account for one-half to two-thirds of the total gold produced world wide (Joseph, 1984).

Gold may be found in any or combination of the these forms:

(i) Modern placers resulting from weathering due to chemical or thermal conditions;

(ii) Paleo-placer which are mechanically deposited; and

(iii) Hydrothermal vein deposits such as black shales resulting from contemporaneous igneous activity which cause broad gold deposition under a reducing environment. Gold is concentrated in shales as detrital free grains with diameter less than 50μm as precipitates, and in pyrite (FeS_2) or other sulphide minerals. This is the first step in the history of gold placer deposits. Figure 2.1 shows the cross section of a river valley showing the distribution of various classes of placer deposit.

Most alluvial gold deposits occur near the primary gold deposits (gold bearing bed-rock, vein or lode), or a short distance from valleys or benches. Alluvial deposits are formed by mechanical concentration of high density minerals, in moving water, for example streams. Gold is concentrated in favourable areas such as quartzite ridges across streams, plunge pools at the bottom of waterfalls, stream confluences and rapidly flowing stream bends.

2.1.2 Petrography/Ore Microscopy

An intimate knowledge of the mineralogical assemblage of the ore is essential if efficient processing is to be carried out. Not only is the knowledge of the nature of the valuable and gangue minerals required, but also the texture such as aggregation (size), dissemination (distribution) and shape of the minerals within the ore. This is effected by ore microscopy which holds advantage over chemical analysis because the inter-action and combinations of the mineral constituents of an ore or mill product can be visually examined.

Ore microscopy therefore, is useful in predicting the beneficiation requirements of a newly discovered mineral deposit, and is important during its development to indicate needed changes in beneficiation procedures as the

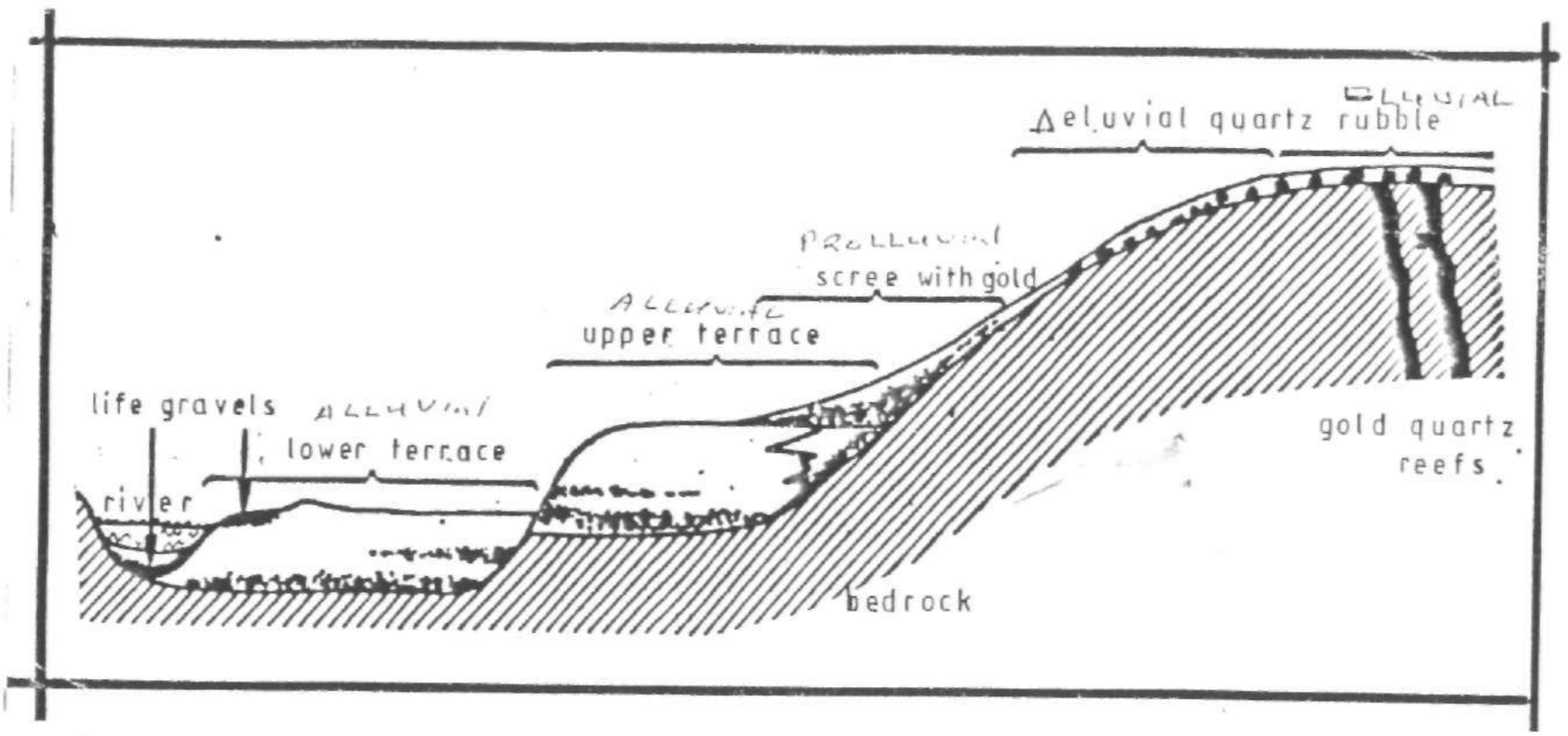


Fig. 2.1 CROSS SECTION OF A RIVER VALLEY SHOWING DISTRIBUTION OF VARIOUS CLASSES OF PLACER DEPOSITS
(after Singoyi and Ndhlovo, 1996)

character of the ore changes laterally or vertically (Hagni, 1978). Ore microscopy has been utilized to obtain a better understanding of the causes of gold losses by the staff of Lake Shore Mine. (Haycock, 1937, Head, 1936; 1937; Henly, 1974; Stallwell & Edwards, 1941; 1946 and Staff, 1936).

2.2 Geology of Ilesha Gold Belt

Ife-Ilesha gold field stretches from Ilesha through Iregun and Osu to the vicinity of Igun (De Swardt, 1947). Gold occurs in two forms which are:

(i) Disseminated gold in quartz-veins occurring in granite gneisses in Iperindo, and

(ii) Alluvial gold associated with amphibolite complex.

The geology of the area was studied by De Swardt (1956) for the Nigerian Geological Survey. De Swardt suggested that alluvial gold is traceable to the wide-spread conformable granular quartz veins. Although these quartz veins show traces of gold, they are too low in content to be of economic significance. Sulphides (Pyrite (FeS_2), arsenopyrite (FeAsS), nickelliferous pyrrhotite ($NiFe_{1-x}S$) have been reported to occur in association with gold, or as disseminations in the amphibole, although no massive sulphide mineralization has been discovered. Occurrence of molybdenite (MnS_2) flakes has also been reported. Talcose rocks (talc: $3MgO.Si_4O_{10}.H_2O$) occur as lenses in the large amphibole bodies. Cassiterite (SnO_2) and columbite $(Fe, Mn)_2O_6$ have also been won along with alluvial gold.

Between 1970-1972 and in 1977 students of Geology Department, University of Ife (now Obafemi Awolowo University) carried out independent geological mapping programmes which covered sheet 243, 1:50,000, Ilesha S.W., S.E.) and sheet 263, 1:50,000 Ondo, N.W. These studies presented generalised information on the petrography, structure and petrogenesis of the major rock units in the area.

Metals and Minerals (Nig.) Ltd. in conjunction with Geology Department, University of Ibadan in 1976 were commissioned by the Nigerian Mining Corporation to carry out a geochemical reconnaissance survey of the Ilesha gold field. The objective was to prospect for gold and other associated metals. A total of 989 active stream sediment were collected from both the amphibolite complex and the biotite schist areas. The results were samples qualitatively interpreted with the major conclusion that no particular element was identified as the pathfinder for gold mineralization. It was therefore suggested that gold is its own indicator and its restricted nature of dispersion hinder the use of conventional geochemical reconnaissance survey.

Elueze (1977), in addition to rock geochemical studies also carried out interpretation of 300 active stream sediment samples selected from the above work. The samples were analysed for 14 trace elements and 4 major elements. His conclusion was that visual interpretation of single element distribution patterns for As, Au, Ni, Cu and other trace elements do not show any clear-cut relationships with known areas of mineralization. This feature is attributed to the enhanced variable background values in the various underground rock. Some metamorphic mineral assemblages in the Ife-Ilesha schist belt according to Elueze (1977) and Cooray (1976) is shown in Table 2.1

2.3 Gold Extraction Processes

Methods of gold extraction depend on physical and chemical properties of the gold and those of its associated minerals. Concentration of gold utilizes the following properties:

1. The high relative density of gold (19.3 when pure) normally between 15 - 19 depending on the amount of the alloy elements mainly silver.
2. Wetting of gold by mercury in the presence of water to form amalgm.
3. The solubility of gold in weak cyanide (alkaline) solutions.

TABLE 2.1

SOME METAMORPHIC MINERAL ASSEMBLAGES IN THE IFE-ILESA SCHIST BELT

(After Elueze, 1977; Cooray 1970)

ELUEZE (1977)			COORAY (1970)		
ROCK GROUP	MINERAL ASSEMBLAGES	TYPE	ROCK GROUP	MINERAL ASSEMBLAGES	TYPE
Metasediments	Qtz-Muscovite-chlorite	Greenschist	Pelitic	Quartz-biotite-staurolite	Amphibolite
	Garnet-biotite-Quartz	Amphibolite		Quartz-Almadine-sillimanite	Amphibolite
Gneissic	Quartz-microline-Plagioclase-biotite-(muscovite)	Amphibolite	Qtz-Feldspathic	Quartz-Almadine-plagioclase Quartz-biotite-microcline-plagioclase Quartz-plagioclase-biotite	Amphibolite
Amphibolite-Complex	Epidote-hornblende-Microcline Garnet-hornblende-Quartz	Amphibolite	Basic	Plagioclase-hornblende-garnet (quartz) Plagioclase-Actinolite-Quartz Plagioclase-hornblende-epidote (quartz)	Amphibolite
	Talc-tremolite-chlorite	Greenschist	Magnessian/ Ultrabasic rocks	Talc-anthophyllite Talc-Tremolite Actinolite-garnet	Greenschist

4. The excellent flotability of gold either alone or when contained in sulphides.

Locating and developing new gold deposits, coupled with the need to improve operating margins of existing plants, have prompted much interest in innovative gold processing techniques.

Metallurgists have developed various processing methods and equipment for gold ore treatment that reflect difference in ore characteristics, grade, mineralogy, climate and location. These methods are being applied to free gold, nonrefractory and refractory ores (Pizarro and Schlit, 1984). Typical process flow-chart for gold extraction are shown in Figures 2.2, 2.3 and 2.4.

The preferred processing technique for a particular ore is likely to be the one with the lowest capital and operating costs, commensurate with both speed of processing and high percentage recovery. There are a number of processing steps and they include the following:

(1) Gravity concentration

  (a) panning

  (b) jigging

  (c) centrifugal concentrator

  (d) bambazonke sluice box

  (e) sluice box

  (f) shaking table.

(2) Gravity concentration with amalgamation, retorting and smelting

(3) Flotation concentration methods

  (a) Froth flotation

  (b) Coal-gold agglomeration

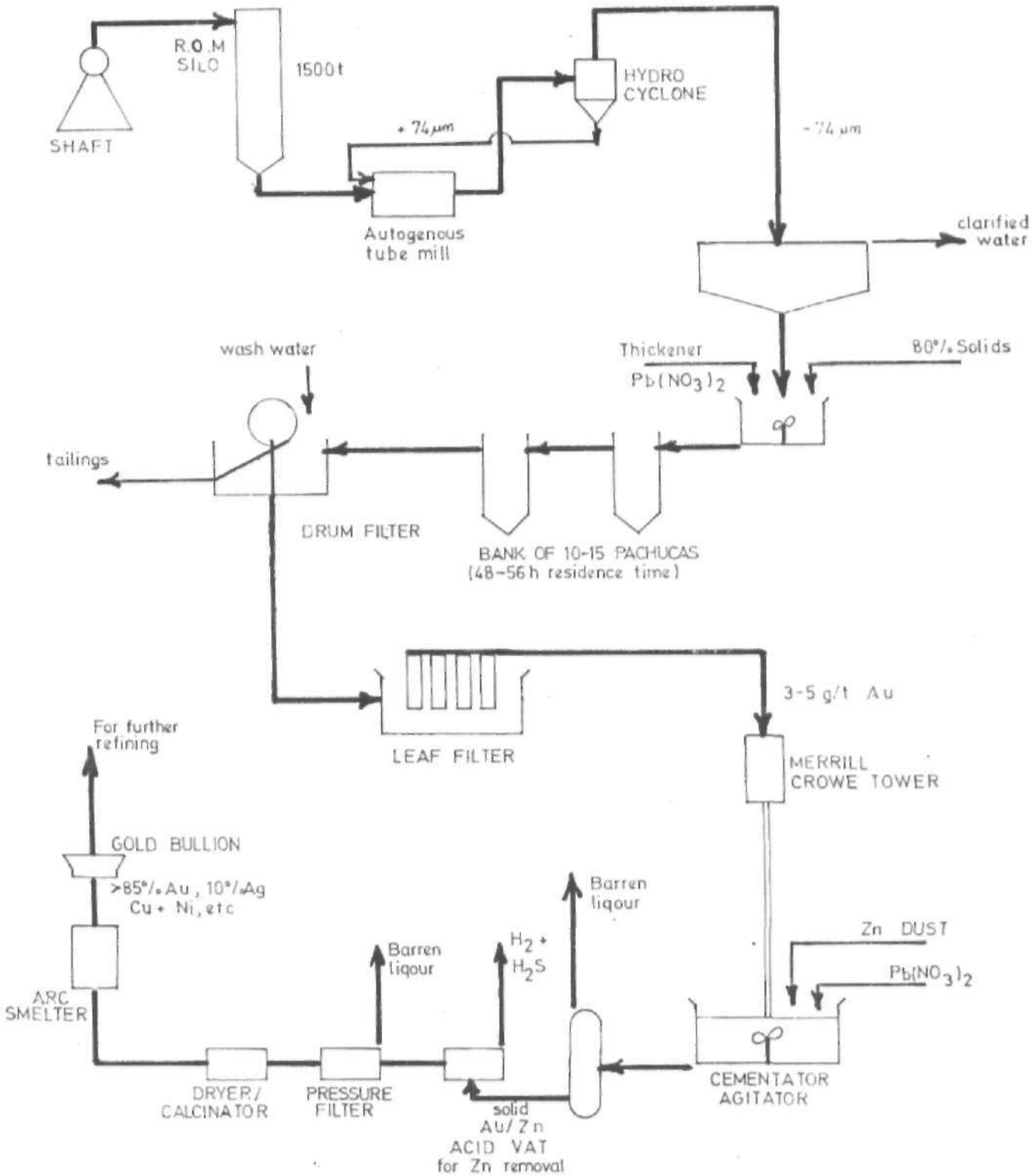


Fig.2.2 A typical Hydrometallurgical process scheme for extraction of fine Gold.
(SOURCE ; ANDREW/ BANDA, 1995)

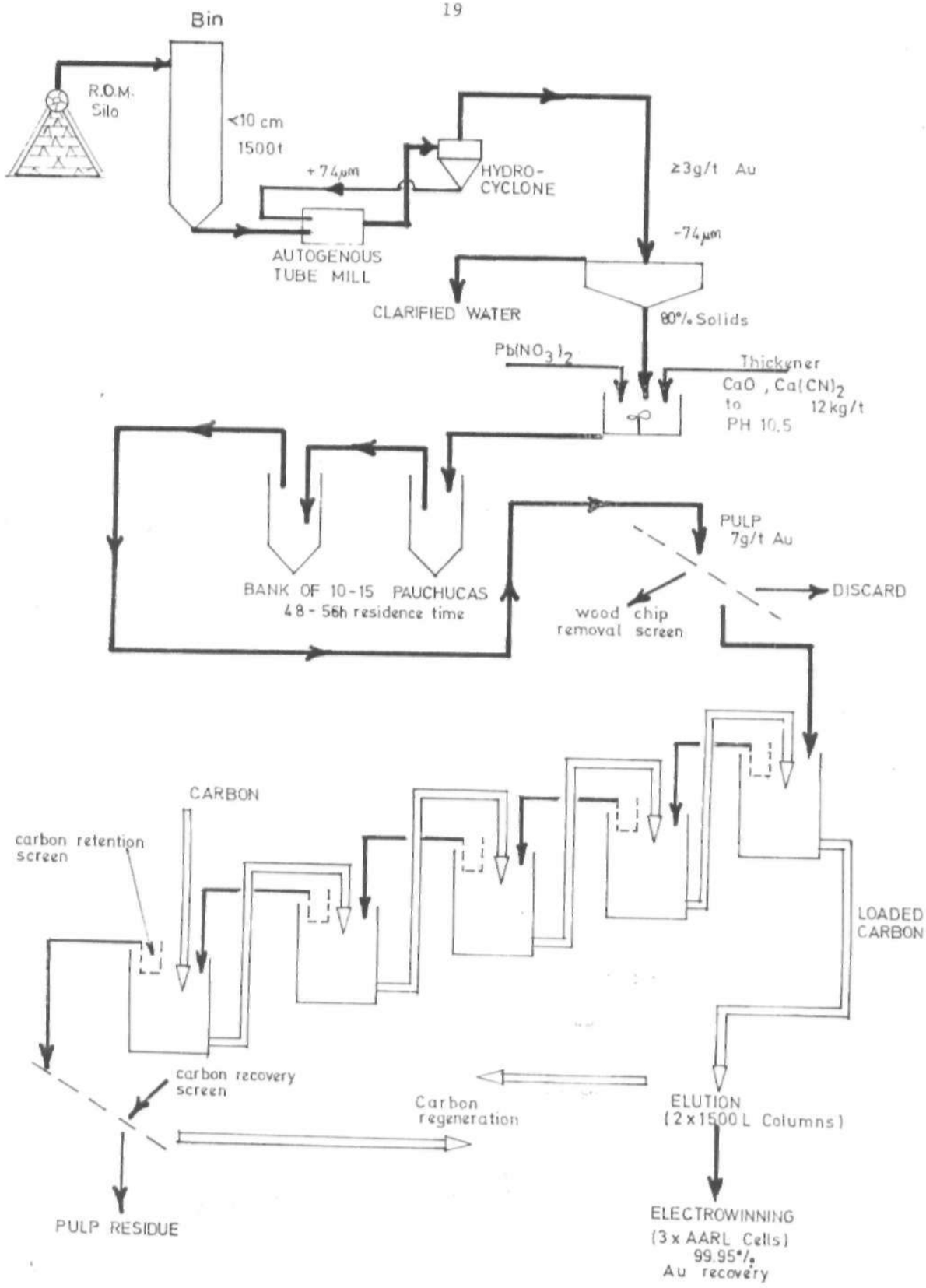


Fig.2.3 A typical Carbon in Pulp process flowsheet for Gold extraction.
(SOURCE : ANDREW BANDA , 1995)

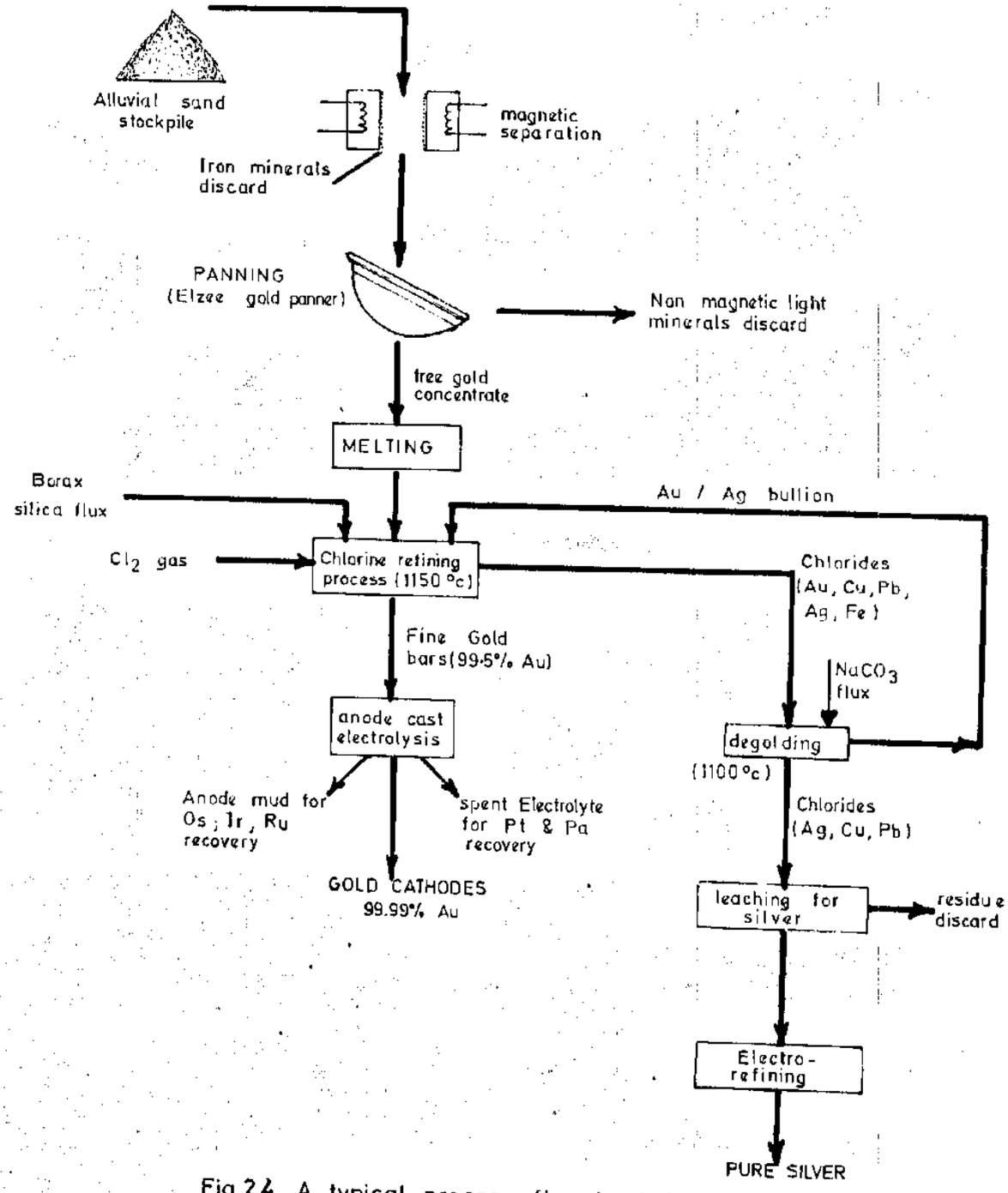


Fig.2.4 A typical process flowsheet for extracting and refining free Gold.
(SOURCE ; ANDREW BANDA, 1985)

(4) Hydrometallurgical processes which entails leaching and gold recovery processes:

(a) Leaching processes:

(i) Agitation leaching, including pressure leaching

(ii) Bacterial leaching

(iii) In-situ leaching

(iv) Percolation leaching

(v) Heap leaching

(vi) Thiourea process.

(b) Gold recovery processes from Pregnant Solution:

(i) Cementation - Merrill-Crown process

(ii) Carbon in pulp

(iii) Resin in pulp

(iv) Electrowinning

(v) Ion exchange

(vi) Solvent extraction

The amenability of these extraction techniques to the three types of gold ores - free gold, non-refractory and refractory ores- are discussed below:

(i) Free gold ores: If the gold ore in the alluvial deposit is free gold and coarse, simple gravity concentration methods such as panning, jigging, tabling, sluicing and centrifuging may be effective. If the free gold in the alluvial deposit is too fine for gravity concentraiton methods to be effective, a combination of gravity separation and amalgamation may be used.

(ii) Non-refractory ores: These are readily leachable gold ores. If the gold occurs as very fine dissemination within the host rock a hydrometal-

lurgical extraction process may be effective for gold extraction. the gold hydrometallurgical Non-refractory ores are amenable to in-situ leaching, heap leaching, percolation (vat) leaching, agitation leaching (including pressure leaching) and leaching by thiourea.

(iii) Refractory ores: Refractory ores are problematic ores. They include chemically combined gold (tellurides), free gold locked in refractory materials (especially pyrite and arsenopyrite) and leachable gold that is reabsorbed on carbonaceous matter in the ore. When gold is locked in or associated with sulphides, processing generally begins with flotation concentration. However, the sulphides or arsenides must generally be oxidised before recovery and this is effected by roasting the flotation concentrate, and then cyaniding the cinder.

The current trend is to use hydrometallurgical methods (such as pressure leaching and bacterial leaching).

2.3.1 Gravity Concentration Methods

In gravity concentration methods, the difference in the specific gravity between the mineral(s) and the gangue is used to separate them. This utilises the differential movement of minerals due to mass effects, usually, in hydraulic current. It has the advantage of producing less environmental pollution. Typical gravity separation methods - bambazonke sluice box, ordinary sluice box, principle of table action are shown in Figures 2.5, 2.6 and 2.7 respectively.

The centrifugal separators combine the effect of specific gravity difference of the mineral(s) of the gangue, and that of centrifugal force of separating medium to produce the concentrate.

The centrifugal separator (Figure 2.8) has become popular over the last five years because of its ability to recover fine gold particles. This machine

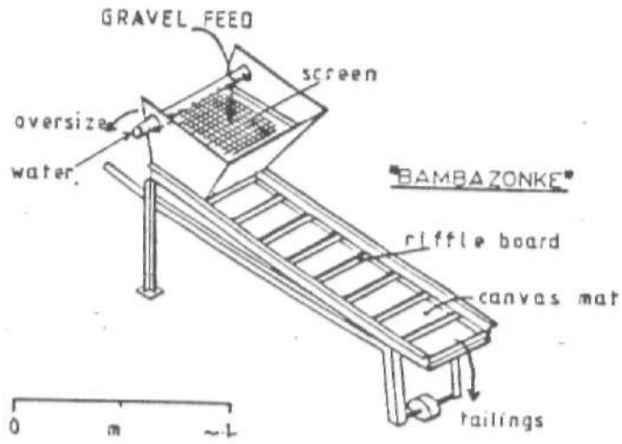


Fig. 2.5 Bambazonke sluice box
(Recommendable efficient machine)

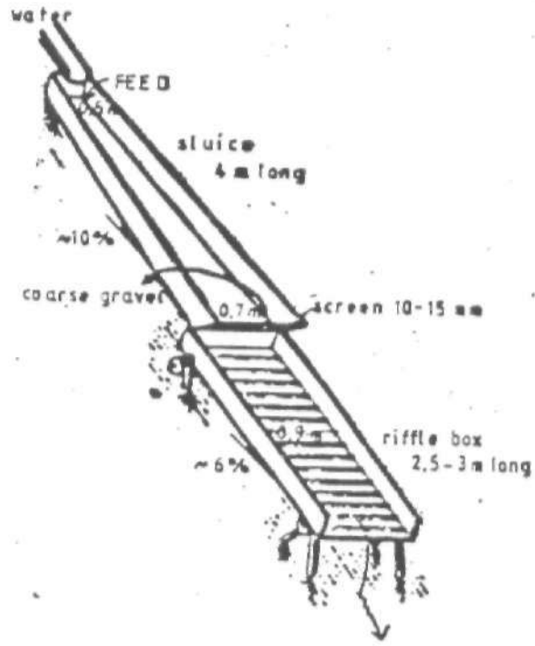


Fig. 2.6 Sluice box

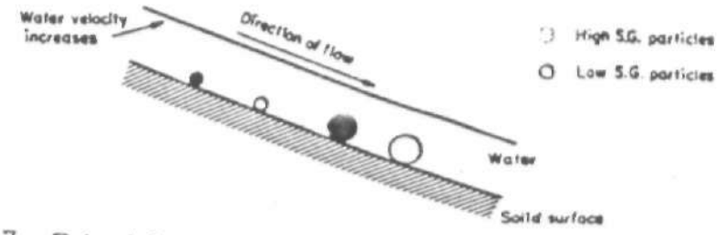
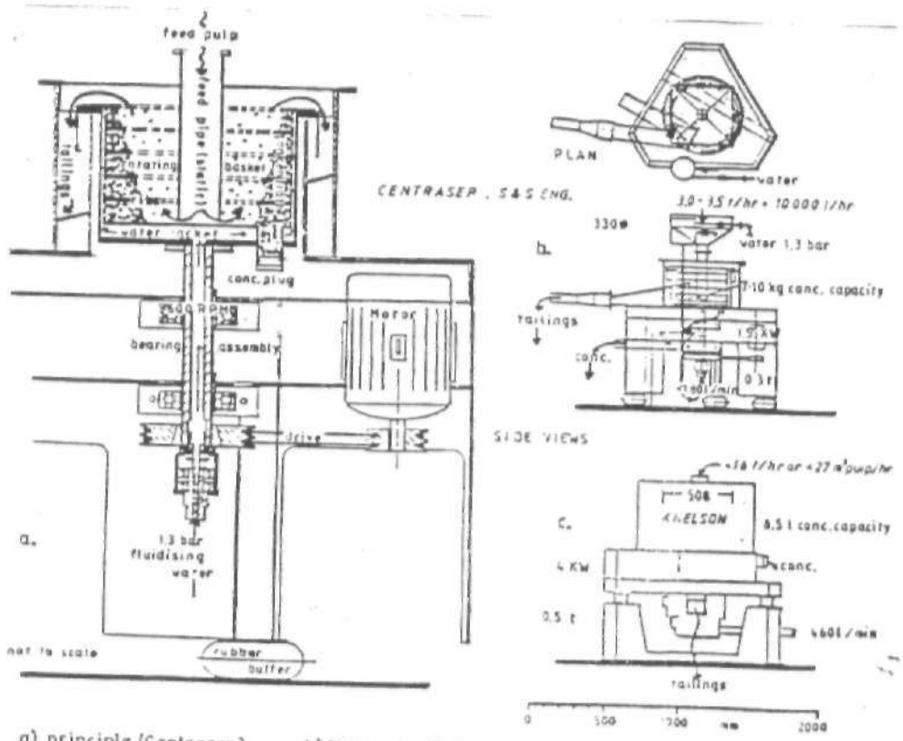


Fig. 2.7 Principle of table action



a) principle (Centrasep). b) 330mm bowl diameter (Centrasep, Zimbabwe).
c) 20" diameter Knelson (Canada) separator

Fig. 2.8 Centrifugal separators

of the future can upgrade low grade concentrate to almost pure gold which can be melted to bullion in a crucible thereby avoiding amalgamation step.

2.3.2 Amalgamation

Before the invention of cyanidation, amalgamation was the most important process for recovery of gold. This process involves the treatment of gold concentrate with liquid mercury. As a result, gold is dissolved in mercury forming a gold-mercury alloy known as an amalgam which can be trapped and removed from the ore pulp. On heating, the mercury volatilises and impure gold bullion is left. Alternatively, the gold is recovered from the amalgam by treating it with concentrated nitric acid. Gold and mercury are known to form two compounds: Au_2Hg and $AuHg_2$ both with a peritectic reaction at 410^0C and 310^0C respectively (Jain, 1987).

Amalgamation is a relatively simple process but has a lot of drawbacks which include the following:

(1) Mercury is very expensive. It is even a controlled metal, so there is no guarantee that one can always get it when it is needed;

(2) There is the problem of amalgamation inhibitors which interfere with amalgamation reaction. These include substances dissolved in water such as alkali sulphides, undissolved or suspended substances such as sulphide minerals (particularly those containing antimony and arsenic) and organic matters. These decrease the wetability of gold by mercury and also tarnish the gold.

(3) Gold recovery is very low if the gold contained in the ore is not free. It was reported that gold was won by passing the liberated ore over gold plate coated with mercury. It extracted the precious metal with an efficiency of between 75 and 80 per cent. When pyrite ore was encountered, the recovery fell below 50 per cent (Cowey, 1994).

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(4) Mercury is an environmentally hazardous substance which has been linked with major human disease epidemics (Muyanda, 1996).

2.3.3 Flotation Concentration Methods

This is a physico-chemical method of separation of minerals from their gangues, utilising difference in surface properties of the mineral.

Froth flotation, which is becoming the most important method of concentration, is based on the affinity of some minerals for rising air-bubbles within the agitated pulp. By conditioning of the pulp with various reagents such as conditioners and depressants, it is possible to make the valuable minerals air-avid (aerophilic) and the gangue minerals water-avid (aerophobic). This results in separation by transfer of the valuable minerals to the air-bubbles which form the froth, floating on the surface of the pulp.

Froth flotation is a prerogative for sulphide minerals; hence it is used for concentrating gold ores associated with sulphide minerals. Thereafter, the concentrate is roasted to convert it to oxides before the cyanidation process.

A novel approach to gold recovery known as coal-gold agglomeration (CGA) has evolved through the research efforts of the British Petroleum Plc. In this process, liberated gold grains are recovered with high efficiency by adhering to the agglomerates of coal and oil. (Manwaring et. al., 1986; Buckely et. al., 1989). Figure 2.9 shows the schematic diagram coal-gold agglomeration flowsheet.

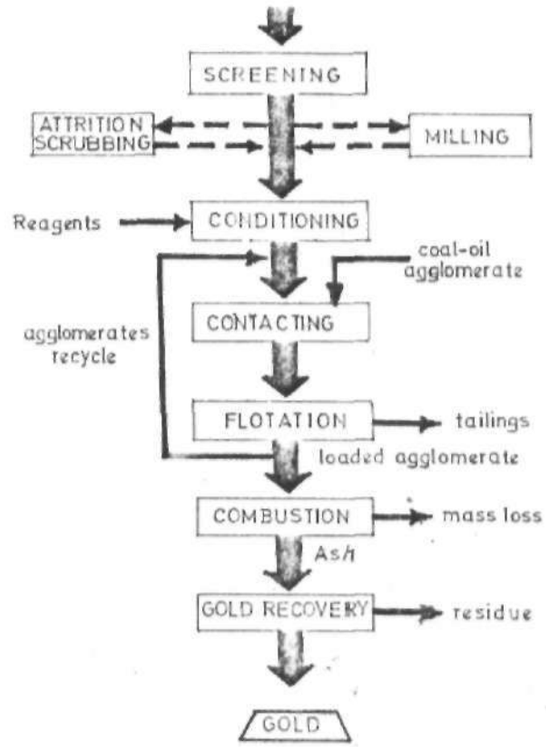


Fig.2.9 Schematic coal Gold agglomeration flowsheet.
(Marwaring et al., 1986; Buckely et al., 1989)

2.3.4 Hydrometallurgical Processes and Theories on Gold Dissolution in Cyanide Solutions

Hydrometallurgy entails two distinct processes.

(1) Leaching: Getting the desired metal or processed metal/mineral from the ore or from other metallurgical products into solution.

(2) Getting the desired metal out of solution.

The inventors of cyanidation were MacArthur and Forrest (1887). Since 1887 the process has spread to every gold and silver mining district in the world. Cyanidation (Jain, 1987) process for gold recovery was first used in 1894 to recover gold from the ores containing gold in the form of sulphide, which did not respond to amalgamation. This has been used later to recover gold which responds poorly to gravity concentration or flotation concentration. It is only of recent that Soviet metallurgist Plaksin and Kozhukokhova (1940, 1960) showed interest in substituting sodium cyanide by a non-toxic reagent, thiourea ($NH_2.CS.NH_2$), which is reported to have extracted more gold than cyanide in the presence of oxygen. However its commercial use is still in the nascent stage.

Hydrometallurgical technique for gold extraction consists of the following stages:

(i) Preparation of ore to expose its gold values;

(ii) Leaching of gold ores to dissolve the gold values;

(iii) Separation of solids from pregnant solution;

(iv) Clarification and de-aeration of solution;

(v) Gold precipitation;

(vi) Treatment of the precipitated gold.

Many theories have been put forward to explain the mechanism of dissolution of gold in cyanide solutions. Comprehensive coverage of the chemistry of cyanidation process has been provided by Finkelstein (1922) and Barskey *et al* (1934). They all agreed that the overall dissolution of gold in dilute cyanide solution may be represented by the classic Elsner's equation (Elsner, 1846).

(i) Elsner's oxygen theory. Elsner (1846) was the first to recognize that oxygen was essential for the dissolution of gold in cyanide solutions; and he represented the reaction as:

$$4Au + 8CN^- + O_2 + 2H_2O \rightarrow 4[Au(CN)_2]^- + 4OH^- \quad (1).$$

(ii) Bodlander's hydrogen peroxide theory: Bodlander (1896) suggested that dissolution proceeds in two stages, according to the equations;

$$2Au + 4CN^- + O_2 + 2H_2O \rightarrow 2[Au(CN)_2]^- + 2OH^- + H_2O_2 \quad (2)$$

$$2Au + 4CN^- + H_2O_2 \rightarrow 2[Au(CN)_2]^- + 2OH^- \quad (3)$$

in which hydrogen peroxide is formed as an intermediate product. He supported his view by being able to detect H_2O_2 in the solution. The overall equation for these two steps is Elsner's equation (equation 1). The above reactions clearly indicate that the leaching mechanisms involved in the cyanidation process is electrochemical in nature as was initially postulated by Kudryk and Kellog (1954).

(iii) Corrosion theory. Boonstra (1953) was the first to recognise that the dissolution of gold in cyanide solution is similar to the metal corrosion process. Oxygen dissolved in the solution is reduced to hydrogen peroxide and the hydroxyl ion.

He pointed out that Bodlander's equation should be further divided into the following steps:

$$O_2 + 2H_2O + 2e \rightarrow H_2O_2 + 2OH^- \quad (4)$$

$$H_2O_2 + 2e \rightarrow 2OH^- \quad (5)$$

$$Au \rightarrow Au^+ + e \quad (6)$$

$$Au^+ + CN^- \rightarrow AuCN^- \quad (7)$$

$$AuCN^- + CN^- \rightarrow Au(CN)_2^- \quad (8)$$

This was demonstrated later by Thompson.(1947)

(iv) Electrochemical theory. This is the most recent theory reported in the literature. Habashi (1966, 1967) showed that the dissolution process, which is electrochemical in nature, are described by the following reactions:

Anodic reactions:

$$Au \rightarrow Au^+ + e \quad (9)$$

$$Au^+ + CN^- \rightarrow AuCN \quad (10)$$

$$AuCN + CN^- \rightarrow [Au(CN)_2]^- \quad (11)$$

Cathodic reactions:

$$O_2 + 2H_2O + 2e \rightarrow H_2O_2 + 2OH^- \quad (12)$$

$$H_2O_2 + 2e \rightarrow 2OH^- \quad (13)$$

Thus the overall reaction is given as:

$$2Au + 4CN^- + O_2 + 2H_2O \rightarrow 2[Au(CN)_2]^- + 2OH^- + H_2O_2 \quad (14)$$

However, experiments show that the dissolution of gold in NaCN + H_2O_2 in the absence of oxygen is a possible reaction but a slow process. Thus the reaction,

$$2Au + 4NaCN + H_2O_2 \rightarrow 2NaAu(CN)_2 + 2NaOH \quad (15)$$

takes place to a minor extent only. It was observed that the dissolution of gold is inhibited if large amount of hydrogen peroxide is present due to the oxidation of cyanide ion to cyanate ion, which has no dissolving action on the metal, viz:

$$CN^- + H_2O_2 \rightarrow CNO + H_2O \quad (16)$$

Leaching Methods

The grade of the ore, the size of the ore reserve, and the ease with which the mineral values are dissolved in a particular reagent are the controlling factors in the choice of the leaching method. The most common methods of leaching have been listed in section 2.3. This section's focus is on the methods of percolation and heap leaching which are being proposed for the processing Igun gold ore.

Percolation Leaching: Percolation leaching is generally applied to low-grade ores of coarse particle sizes and hence it is referred to as sand leaching (Jackson, 1986). Regularity in the particle size rather than the actual size is the chief factor governing good percolation (Davidson and Duncanson 1977). This method is unsatisfactory if slime is present in the ore.

The method has the advantages of minimum solvent consumption, production of high-grade pregnant solution and the elimination of the expensive thickners and filters.

Heap Leaching: Heap leaching is generally applied to slower leaching, lower grade ores and to higher tonnage deposits. Ore, agglomerated if necessary, is leached on impermeable pads to prevent contamination of near surface aquifers and losses of gold bearing solution (Pizarro and Schillit, 1984).

Cyanide solution is sprayed over the heap via a plastic pipework system distributed over the surface. Leach solution percolates through the ore, dissolves the gold and draines into a channel from which it moves into a large pregnant-solution collecting pond. Permeability, solution delivery and solution recovery are the critical factors in dump or heap leaching of gold (Lastra and Chase, 1984).

During the 1970s the U.S. Bureau of Mines investigated a particle agglomeration technique for improving the flow of leaching solution through heaps of clayed or crushed, low-grade gold-silver ores. This technology which is cost effective because of decreased leach times and improved precious metal recoveries has been adapted on a broad scale by the precious metal processing industry. (Heinen et. al. 1979; McClelland et. al., 1983; McClelland et. al. 1983).

Merrill-Crowe Cementation Process

Recovery methods are itemised under Section 2.3 but our focus will be on Merrill- Crowe cementation process being proposed for the recovery of Igun gold ore. This method is more selective and relatively cheaper than electrowinning.

Cementation involves the reductive precipitation of one metal by another, a galvanic corrosion cell being formed.

Consider the standard electrode potential for cementation process for Au-Zn system at 25^0C. Since

$$E^0_{Au^{3+}/Au} > E^0_{Zn^{2+}/Zn}$$

reduction will take place at the gold electrode while at the anode (Zn) oxidation takes palce.

$Zn \rightarrow Zn^{2+} + 2e.$

The cell potential for cementation for Au-Zn system at 25^0C is given by

$E^0 = 1.45 - (-0.763) = 2.213$ V.

The standard free energy change for the net reaction is

$$\Delta G^0 = -ZFE^0cell = -626.5 \text{ Kj mol}^{-1}_{zn} \quad (17)$$

The high negative value of ΔG^0 confirms that precipitation is thermodynamically favourable.

The Merrill-Crowe precipitation reactions of gold from cyanide solution using zinc powder is represented by the equation:

$$2Au(CN)_2^- + Zn = 2Au + Zn(CN)_4^{2-} \quad (18)$$

Now that CN^- ions are involved, the two electrode systems are given as:

$$Au(CN)_2^- + e = Au + 2CN^-;\ E^0 = -0.67\ V \quad (19)$$

and

$$Zn(CN)_4^{2-} + 2e = Zn + 4CN^-\ ;\ E^0 = -0.26V \quad (20)$$

From where we obtain:

$$E^0_{cell} = -0.67 - (-1.26) = 0.59V \quad (21)$$

It is seen that the standard potentials of these complex cyanide system in equation (20) are much more negative than the corresponding cathodic systems. The negative shift is more marked in the case of gold because of the high stability of the cyanide complex ($\log \beta_2 = 36.0$) as would be expected from the equation:

$$E_c = E^0_{aq} - \frac{RT}{ZF} ln\beta \quad (22)$$

The resulting cell potential for cementation is 0.59V as shown in equation (21) compared with the theoretical 2.21V based on cathionic specie. Thus, the selectivity of precipitation is high and contamination is very low.

In general, the higher the cell potential of cementation reaction the greater is the likelihood of impurity precipitation (Jackson, 1986).

2.4 Previous Work on Ilesha Gold Ore

A lot of sampling/assay work to determine the gold content of Ilesha gold deposit especially on the primary deposit has been carried out. Although there are erratic small pockets of high grade mineralization, averaging 60g/ton, ore grade is generally low (Figiel, 1959). It was observed that although the mill feed on the primary deposit of Ilesha gold ore after crushing to -35 mess Tyler carried up to 22.5dwt/ton (34.875 g/ton), only 5 dwt/ton (7.75 g/ton) was recovered, and the rest 17.5 dwt/ton (127.25 g/ton) was lost in the tailings. The tailings from the Holman blanket table was deslimed and leached for 48 hours with cyanide. An examination of the concentrate revealed that the majority of the gold was extremely fine, which explained the high losses. What happened to the blanket table concentrate and the pregnant solution was not reported.

Flutcher and Clarke (1961) observed that a large proportion of the gold is very fine and probably locked up in an associated sulphide. Hence they indicated that they belong to the category of low-grade mineralized quartz-veins.

Quite recently, Adetunji (1991) investigated the efficiency of yields (from Ilesha alluvial gold concentrate) from amalgamation and cyanide solutions from analytical grade of NaCN and cassava. It was observed that the yields from amalgamation process were lower than yields from cyanide solutions - both standard grade analytical NaCN and cassava cyanide.

Thus far, there is no available literature on the process design for Ilesha gold ore from the time of location of the deposit to the recovery /extraction stage. Hence the relevance of this study on Igun gold deposit ore which is aimed at enhancing gold recovery, reducing production costs and assuring environmental protection. The object of this investigation is to come up with a report proposing a process flowchart for efficient recovery / extraction of gold from Igun gold ore.

CHAPTER THREE

EXPERIMENTAL PROCEDURE

This chapter describes the mineral processing and the mineralogical investigation of Igun alluvial gold deposit. This is followed by analytical procedures - comprising fire-assay-gravimetric method, and the atomic absorption spectrophotometer instrumental analysis - and then by amalgamation and percolation leaching.

3.1 Mineral Processing and Mineralogical Investigation

3.1.1 Pitting, Sampling and Concentration

At Igun, samples were taken by shovels along river channels and concentrated by sluicing and panning. The heavy mineral (black sand) was despatched to the laboratory at Osu near Ilesha where a qualitative test for the presence of gold was conducted. This was proved positive for all samples.

Pits of 2.8 metres diameters were then sunk at various depths, the average depth being 3.5 metres. The wash from each pit was carried headpan by headpan and fed into the scrubber attached to a jig. The average weight of one headpan of wash was 31.89 kilograms (kg). The wash constituent of the alluvial deposit was ascertained and the result is presented in Table 4.1.

A scrubber was used to disintegrate and deslime the run of mines and the product was moved into a jig, where light minerals were separated from heavy ones. The light minerals (tailings) were passed to the tailings dam while the jig concentrate was sent to the shaking table at Osu for another round of gravity separation. The shaking table concentrate was sent to the magnetic separator which separated the magnetic minerals (such as ilmenite and magnetite) from non-magnetic minerals which contain gold. The simplified flowsheet for production of alluvial gold concentrate from the wash is

shown in Figure 4.1.

3.1.2 Particle Size Analysis

Particle size analysis at separation stage is carried out to determine the optimum size of feed to the process for maximum efficiency. Also, it determines the size range at which losses may occur in a mineral processing plant, with a view to reducing the losses.

In order to achieve these objectives for Ilesha gold deposit, half of a headpan of gold concentrate was weighed and then sieved by a vibrating shaker, whose operating data were as follows: Power = 180 watts, Voltage = 220 - 240 volts, Current = 2.25A, Frequency = 50 hertz.

The grade of test sieves used were as follows: (i) 2.00mm (2000μm) (ii) +850μm (iii) +600μm (iv) +300μm (v) + 150μm (vi) + 75μm (vii) - 75μm The result of the particle size analysis is contained in Table 4.2.

3.1.3 Petrography

Slides prepared from briquettes of the various particle sizes were made and examined under the petrographic microscope - under plane polarized light and cross nicols. The resulting photomicrographs are shown in Plates I - XIV. The result of the modal analysis emanating from the ore microscopy are presented in Table 4.3.

3.1.4 X-ray Diffractometry

For mineral identification and structural analysis of minerals in the Igun gold ore, x-ray diffraction analysis of the sample was carried out on the seven particle size fractions obtained from sieve analysis. These particle sizes were subjected to x-ray diffractometry using the old model of PIN 1410 x-ray diffractometer at the school of Mines of the University of Zambia in Lusaka using Cu-Kα radiation. The d-spacings of Igun gold oncentrate samples were computed for Cu K-alpha λ=1.54179A^0 using the Bragg equation: $n\lambda = 2d\sin\theta$.

The minerals identification was then effected with the aid of a box file (see Appendix 1).

3.2 Analytical Procedures

Before designing the extraction process for gold ore, it is necessary to determine the gold content of the ore by quantitative analysis. Whether qualitative or quantitative, analysis are based on characteristics, properties and mode of occurrence of gold. Gold's general properties and various gold tests are given in Appendix 3. Measurements of gold purity, conversion factors and strategic importance of gold are contained in Appendix 4.

Seven samples of different particle size fractions including that of the unsized concentrate from Igun in Ilesha area of Nigeria were taken to the Department of Metallurgy and Mineral Processing, School of Mines of the University of Zambia in Lusaka. Samples were subjected to fire-assay-gravimetric analysis. This was followed by instrumental analytical procedure using Atomic Absorption Spectrophotometer (AAS) for confirmation.

3.2.1 Analysis of Gold by Fire-Assay-Gravimetric Method.

Seven sets of gold ore concentrate samples from grain size range of +2000μm to -75μm were taken to the Geological Survey Department of the Ministry of

Mines and Minerals Development of the Republic of Zambia for gravimetric-fire-assay test. Result is contained in Table 4.3.

50 grammes of each of the particle size fraction were used. Each was mixed with 175gm of flux No.2 whose composition by weight is as follows: PbO = 61%; $NaCO_3$ = 33%; Borax glass = 3.4%; Flour = 2.6%;

The fluxed samples were heated in the furnace to a temperature of 1100^{0}C for 60 minutes to obtain lead bullion bearing the gold and the siliceous glass bearing the slag. The lead bullion were cupelled in a bone ash at a temperature of 960^{0}C for 45 minutes to obtain gold prils. The prils were boiled in nitric acid for 20 minutes and were then annealed in a porcelain dish for 15 minutes.

The prils were weighed on sensitive 5 decimal point scale to get the assay value. The result of the investigation is contained in Geological Survey Department (Ministry of Mines and Minerals Development, Republic of Zambia) Laboratory Report Lab. Ref. No. M95/8 dated 18/03/96 shown in Appendix 7 and in Table 4.4.

3.2.2 Analysis of Gold in Ore Sample by AAS

Seven samples of different particle sizes ranging from 2000μm to -75μm weighing 12g each were mixed with 100ml hydrogen bromine solution and left overnight.

The aliquot was boiled to expel the excess Br_2 and diluted to 200ml with distilled water and filtered through No.4 Whatman filter paper.

The filtrate was put in a separating flask (250ml.) to which 10ml of Isobutylmethyl ketone ((MIBK) was added. Then a stopper was put on the flask and shaken for 2 minutes.

Next was the separation of the phases into the ketone and aqueous phases and the retention of both. 10ml of MIBK were added to the aqueous phase, and further extraction took place with the aqueous phase discarded.

The ketone phases were combined and washed twice by extracting with successive 25ml volume of 2M hydrobromic acid.

The aqueous phase was once again discarded and the ketone phase was diluted to 50ml with MIBK.

Gold was taken into solution by the bromine and hydrobromic acid, and the MIBK extracts the gold as bromoauric acid. The ketone phase was washed with acid to remove Fe, some of which is extracted with gold.

The AAS was calibrated with standards that have been freshly extracted by MIBK from 2M HBr, and the ketone phase was aspirated into the flame of AAS. The result of the ore analysis was obtained by computer-operated regression analysis as shown in Table 4.5. Calibration tables used to interpolate results are shown in Appendix 8. Percentage gold determination, gold content in grammes, AAS detection level and AAS sensitivity are given in Appendix 9.

3.2.3 Analysis of Silver by AAS in Ore Sample

Analysis of other elements which are normally associated with gold were also carried out on Igun gold ore. They include the following: silver, copper, lead and zinc. Silver stands in a distinct class.

Reagents for Analysis of Silver

1. HNO_3, S.G.: 1.42AR

2. 8M HNO_3, Mix 50ml HNO_3 with 50ml H_2O

3. Mercuric nitrate solution (0.03g Mercuric nitrate in 100 ml of HNO_3).

In carrying out the silver analysis, 0.2gm of Igun gold ore sample was weighed into a 250ml beaker.

10ml of mercuric nitrate solution was added and heated to 90^0C for 1 hour and filtered through No.4 Whatman filter.

Dilution to 50ml in a volumetric flask with distilled water followed.

The AAS was calibrated with suitable standard solutions prepared in 8M HNO_3, and standards and samples were aspirated into the AAS flame.

The parts per million (ppm) of silver was obtained by regression analysis (Table 4.6).

3.2.4 Analysis of Copper, Lead and Zinc Content in the Ore Sample by AAS.

Reagents.

1. HNO_3, S.G. 1.42 AR

2. $HClO_4$, 60% w/W, AR

3. Aqua regia:3 HNO_3, 1 HCl

4. HCl, S.G. 1.15 AR

5. 6M HCl, mix 52.5ml HCl with 47.7ml of H_20

6. 3M HCl, mix 26.65ml HCl with 73.35ml H_20.

The analytical procedure involves heating of 0.5019g to 0.5090g of Igun gold concentrate with 10ml acid mixture in 250ml beaker and evaporated to dryness. The heating was followed with addition of 30ml 6M HCl on

low heat for 5 minutes. The sample was then filtered into 100ml volumetric flasks and volume was made up. The AAS instrument was calibrated with appropriate standard solution of suitable concentration prepared in 3M HCl. The standard and sample solutions were aspirated into the AAS flame and the Cu and Pb contents of the sample were obtained by regression analysis as shown in Table 4.6 while that of Zinc is shown in Table 4.7.

3.3 Methods

Having determined the gold content of Igun gold ore concentrate, samples of the concentrates were subjected to the following extractive metallurgy processes one after the other, namely

1. Amalgamation
2. Percolation leaching.

3.3.1 Amalgamation

Amalgamation tests were carried out at the Mineral Processing Laboratory of the Federal University of Technology, Akure, Nigeria where the following experimental procedures were followed:

87.76g of the concentrate sample of Igun gold ore was subjected to screen analysis. 5g, 10g, 7.5g, 20g, 20g, 2.5g and 0.17g of $+2000\mu m$, $-2000 +850\mu m$, $-850 +600\mu m$, $-600 +300\mu m$, $-300 +150\mu m$, $-150 +75\mu m$ and $-75\mu m$ particle size fractions respectively were added to seven plastic basins which had been tared to zero weights.

Distilled water was added to the seven samples to form pulps and thereafter about 5g of mercury was added to each pulp. The basins were then carefully and skillfully agitated to concentrate the gold values

at the bottom. The Hg-bead (amalgam so called) was decanted into weighed crucibles (seven of them).

The crucibles containing the amalgam were heated in a furnace to a temperature of 500^0C for 15 minutes, when the mercury had volatilised and they were allowed to cool. The crucible and their contents were then weighed on a sensitive 5 decimal-point scale. The result of amalgamation tests carried out on Igun gold ore is shown in Table 4.8.

3.3.2 Percolation Leaching Method.

Amenability of the ore samples (alluvial deposits) to percolation leaching was carried out at the Department of Metallurgy & Mineral Processing Laboratory of the University of Zambia, Lusaka and the following apparatus/accessories and consumables were used.

Apparatus and Materials.

The major items of equipment used are Vacuum pump and pH meter. The experimental materials include hoses, cotton wool, compressed air gloves and safety kits. The consumables used are essentially sodium cyanide, zinc dust, lime, fluxes (borax, silica sand), sodium hypochlorite, lead nitrate, distilled water and filter paper.

Procedure

The experimental set-up for percolation leaching is shown in Figures 3.1 and 3.2, (See Plate XV in Appendix 6). Sodium cyanide was used to leach 200g of each of the samples. The operating parameters of resident time, pulp hydrogen ion concentration, sodium cyanide concentration and mineral particle sizes were varied while gold recovery in each case was noted. Stoichiometric computations for sodium cyanide concentration based on the cyanidation reaction in equation (14) are contained in Appendix 5.

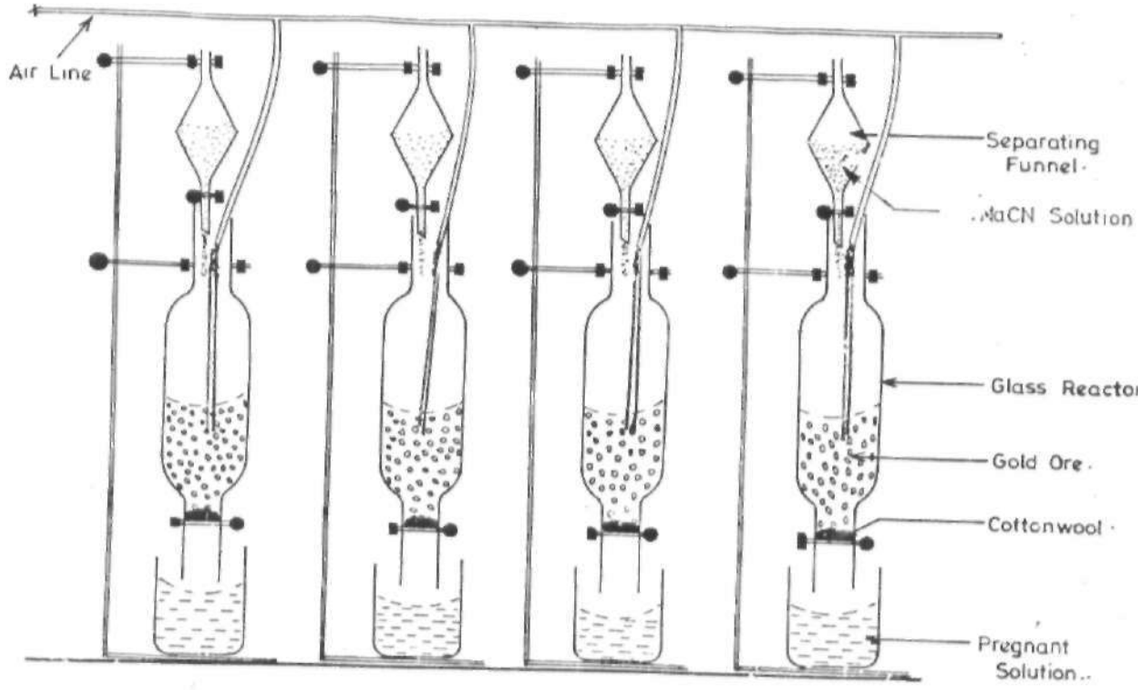


Fig. 3.1 Experimental set-up for percolation leaching

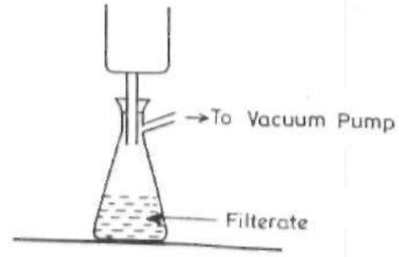


Fig. 3.2 Filteration and Dearation

The unsized concentrate sample was used initially and sized samples were used to test the effect of particle size on gold recovery. Calcium oxide was used to control the pH of the cyanide solution and to help flocculation. Oxygen was supplied into the reaction vessels by a compressed air of 0.5 bar (0.5 $\times 10^5$ Pascals) via a set of hoses. Recirculation of the cyanide solution and the mixing of the gold concentrate was done manually every hour because no recirculating pump was available.

Separation of the pregnant solution and clarification was effected by the use of filter paper (Whatman No.4) inserted into a conical flask connected to a vacuum pump (Figure 3.2). De-aeration of the solution was simultaneously effected by the pump.

Then a prepared solution (0.5g of lead nitrate and 2.5g of zinc dust in 10ml of water) was added to the clarified pregnant solution in order to produce a gold precipitate. But no precipitate was observed perhaps due to the low gold content in the solution. Consequently, the gold in the pregnant solution was extracted into the organic phase and analysed by atomic absorption spectrophotometer (AAS) as followed: The cyanide solution was filtered through Whatman No.4, and 300ml of the filtrate was measured in a 100ml separating funnel. This was followed by the addition of 20ml isobutyldimenthyl Ketone (DIBK) which was shaken vigorously for one minute. 100ml of 20% NaCl was then added. The aqueous phase was drained out and the organic phase was collected in a 25ml volumetric flask. The organic phase was aspirated on AAS using standards previously prepared in organic phase medium.

Effect of Cyanide Concentration on Gold Recovery.

The table below (Table 3.1) summarises the experimental conditions used for gold recovery using unsized concentrate. The concentrations of sodium cyanide were obtained stochiometrically from cyanidation reaction detailedly

given in Appendix 5.

Table 3.1: Experimental Conditions for Gold Recovery Versus Cyanide Concentration

Sample Number	Sample Weight (g)	Sample Volume (ml)	Sample Aliquot Volume (ml)	Cyanide Concentration (%)
1	200	2000	250	0.05
2	200	1000	250	0.10
3	200	500	250	0.20
4	200	333.3	250	0.30

The experimental parameters are stated below:

Retention time = 4hours.

pH = 11.5.

Cyanide concentrations: 0.05%, 0.1%, 0.2%, and 0.3%.

Particle size fraction: Unsized concentrate (+2000μm).

The results obtained are as shown in Table 4.16 in Appendix 10 and graphically in Figure 4.9.

Effect of Retention Time on Gold Recovery.

The table below (Table 3.2) summarises the experimental conditions used for gold recovery versus retention time.

Table 3.2: Experimental Conditions for Gold Recovery Versus Retention Time.

Sample Number	Sample Weight (g)	Sample Volume (ml)	Aliquot Volume (ml)	Retention Time (hr)
1	200	333.3	190	2
2	200	333.3	200	6
3	200	333.3	200	7

The experimental parameters are stated below:

pH = 11.5.

Cyanide-concentration = 0.3%

Retention time = 2 hours, 6 hours, and 7 hours.

Particle size fraction: Unsized concentrate (+2000μm).

The results obtained are as shown in Table 4.9.

Effect of pH on Gold Recovery.

The table below (Table 3.3) summarises the experimental conditions used for gold recovery versus hydrogen ion concentration.

Table 3.3: Experimental Conditions For Gold Recovery Versus pH

Sample Number	Sample Weight (g)	Sample Volume (ml)	Aliquot Volume (ml)	pH
1	200	333.3	200	11.5
2	200	333.3	200	12.0
3	200	333.3	200	12.5
4	200	333.3	200	13.0

The experimental parameters are stated below:

Cyanide Concentration = 0.3%

Retention time = 6 hours

pH values used = 11.5, 12, 12.5 and 13.

Particle size fraction: Unsized concentrate (+2000μm).

The results obtained are shown in Table 4.17 and Figure 4.10.

Effect of Ore Particle Size on Gold Recovery.

The table below (Table 3.4) summarises the experimental conditions used for gold recovery versus particle size.

Table 3.4: Experimental Conditions for Gold Recovery Versus Particle Size

Sample Number	Particle Size in Microns(μm)	Sample Weight (g)	Sample Volume (ml)
1	unsized concentrate	200	333.3
2	-2000 +850	200	333.3
3	-850 +600	200	333.3
4	-600 +300	200	333.3
5	-300 +150	200	333.3
6	-150 +75	200	333.3
7	-75	200	333.3

The experimental parameters are stated below:

Cyanide concentration = 0.3%

Retention time = 6 hours

pH = 11.5

The results obtained are as stated in Table 4.18 and Figure 4.11.

Disposal of Toxic Wastes

After the experiments, filter papers including some with caked slurry on them were collected and burnt.

Due to the poisonous nature of cyanide solutions, these were pretreated before discarding them to the environment.

The aqueous phases were recombined with the remainder of solution and slurry. These were treated by adding 4 grammes of commercial grade sodium hypochloride, resulting in the neutralisation of the cyanide. The cyanide strength was then checked until less than 0.017% NaCN was achieved. Determination of total cyanide in solution as described in Dorr and Bosqui (1950) is as follows:

10ml of potassium iodide in sodium hydroxide solution were added to 25ml of filtered clear cyanide solution. This was titerated with a standard silver nitrate solution to the end point (1st permanent yellow opalescence).

CHAPTER FOUR

RESULTS AND DISCUSSION

This chapter presents the results of mineral processing and mineralogical investigations, ore analysis and the main experimental work. It also covers discussions on mineralogical characterization, process metallurgy of, and flowsheet development for Igun gold ore.

4.1 Results of Mineral Processing and Mineralogical Investigations

The results of mineral processing and mineralogical investigations aspect of the process design for Igun alluvial gold ore commences with the wash constituents of the ore presented in Table 4.1.

Table 4.1 Wash Constituents of Igun Alluvial Gold Ore

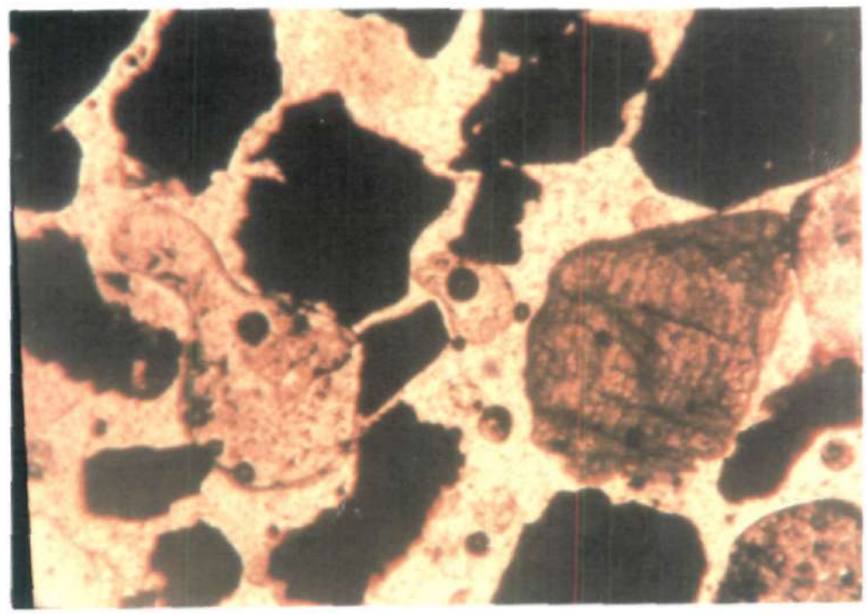
Wash Constituent	Mineral Composition by Weight (g)	Percentage Composition
Clay	17,539.50	55.55
Haematite/Magnetite (magnetic materials)	11,799.30	37.37
Heavy Minerals (Concentrate)	2,232.30	7.07
Total	31,571.10	99.99

Average weight of one headpan of wash = 31.89 kg = 31,890g

4.1.1 Production of Alluvial Gold Concentrate

A simplified flowchart for the production of alluvial gold concentrate from the wash to the unsized gold concentrate is shown in Figure 4.1. Here, the excavated wash runs through scrubbing, jigging, tabling and magnetic separation before the unsized alluvial gold concentrate (black sand) is obtained.

PLATE I



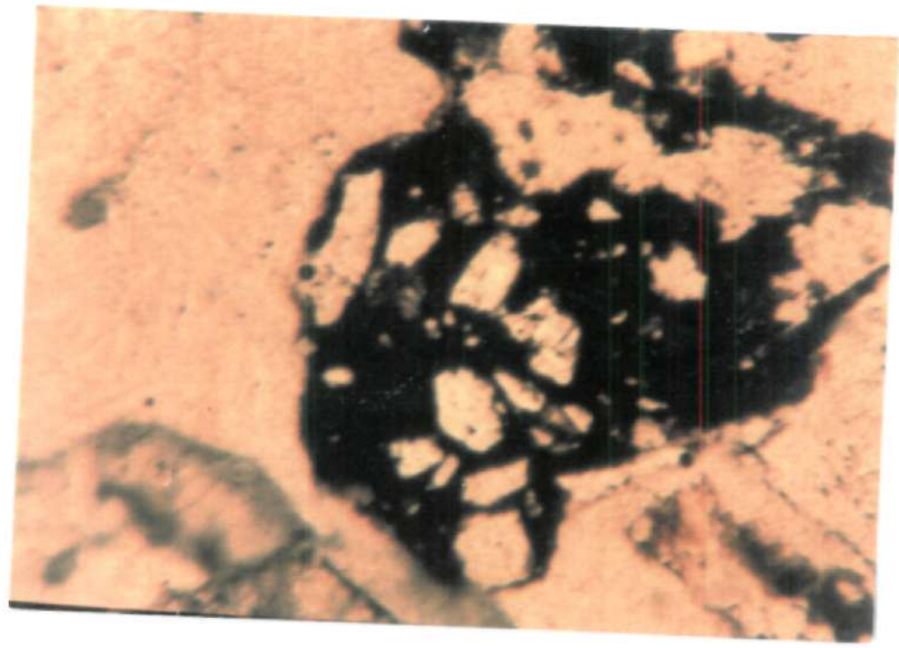
Photomicrograph of the unsized concentrate (+2000μm) of Ikeha Alluvial Gold ore under Plane Polarized Light. (Magnification = 4x)

The concentrate consists mainly of opaque grains (> 70%) which is essentially ilmenite.

Surbodinate amount of transparent minerals can be seen and they are identified as pyrite and chalcopyrite.

The round spots are air bubbles.

PLATE II



Photomicrograph of the -2000 +850μm Particle Size Fraction of Ilesha Alluvial Gold ore under Plane Polarized Light. (Magnification - 40)

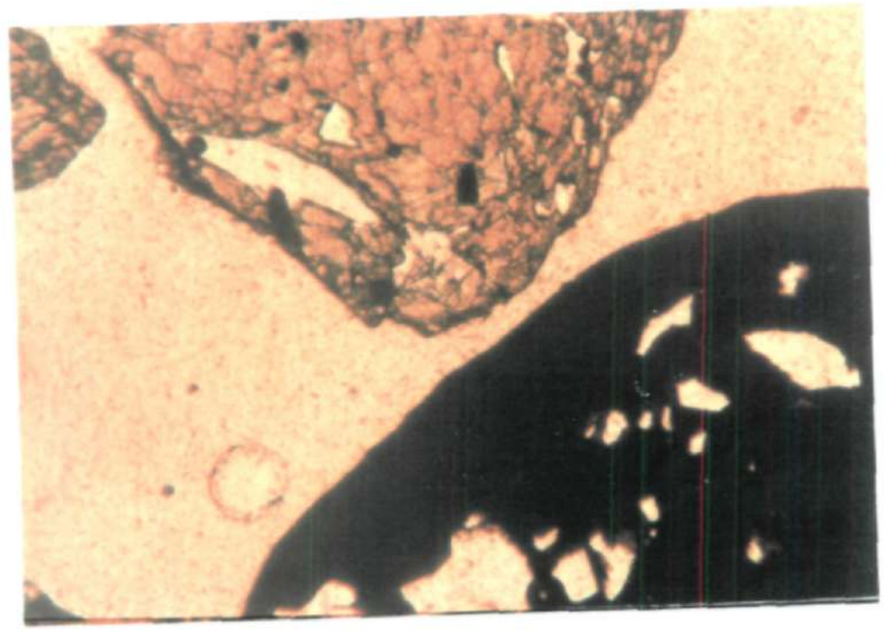
The central massive black phase is iron minerals.

Rusty brown geotite can be observed.

Ilmenite is also present.

The inclusions in the iron matrix include quartz and feldspar.

PLATE III

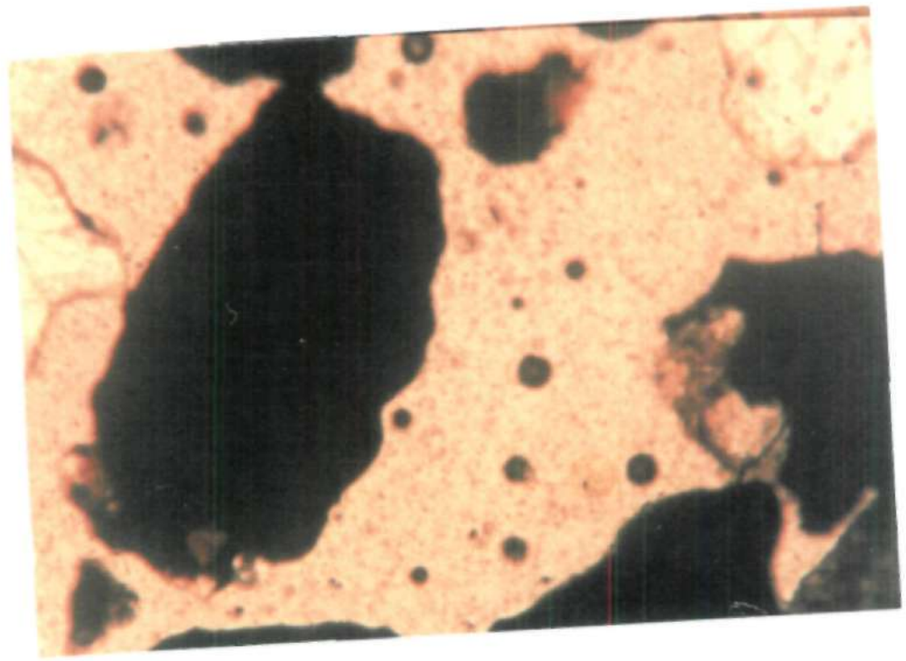


Photomicrograph of the -850 +600μm Particle Size Fraction of Ilesha Alluvial Gold ore under Plane Polarized Light. (Magnification × 40)

The massive black phase in the South East portion of the photomicrograph is essentially ilmenite with quartz inclusion.

At the Northern side is chalcopyrite with inclusions as shiny specks some of which is possibly gold.

PLATE IV



Photomicrograph of the -600 +300μm Particle Size Fraction of Ilesha Alluvial Gold ore under Plane Polarized Light. (Magnification - 40)

The opaque minerals' relative abundance is about 50%.

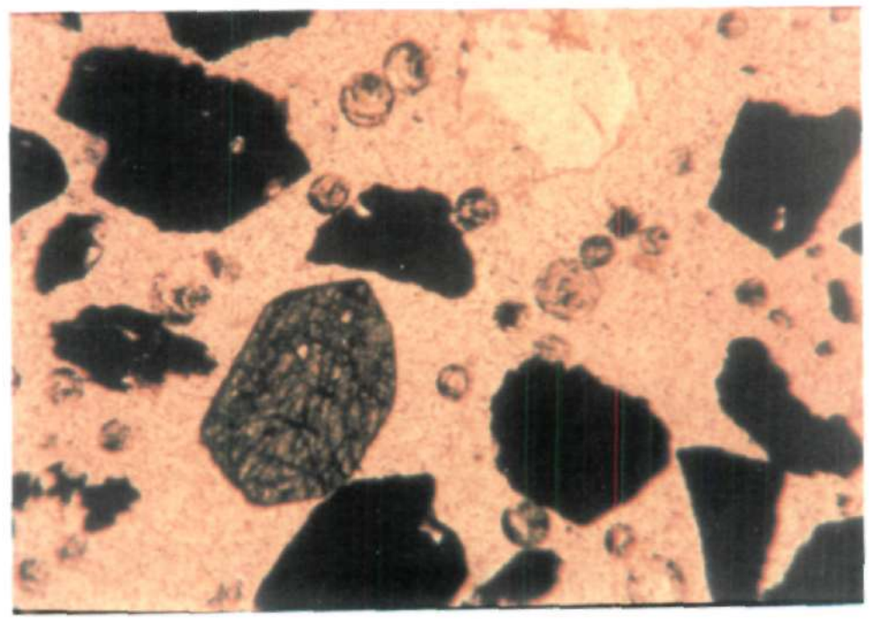
Ilmenite is the most prominent in this portion.

Pyrite and chalcopyrite can be seen.

The rest of the transparent minerals include quartz and feldspar.

The round spots are air bubbles.

PLATE V



The opaque grains within the grandmass is mainly ilmenite.

Round spots are air bubbles.

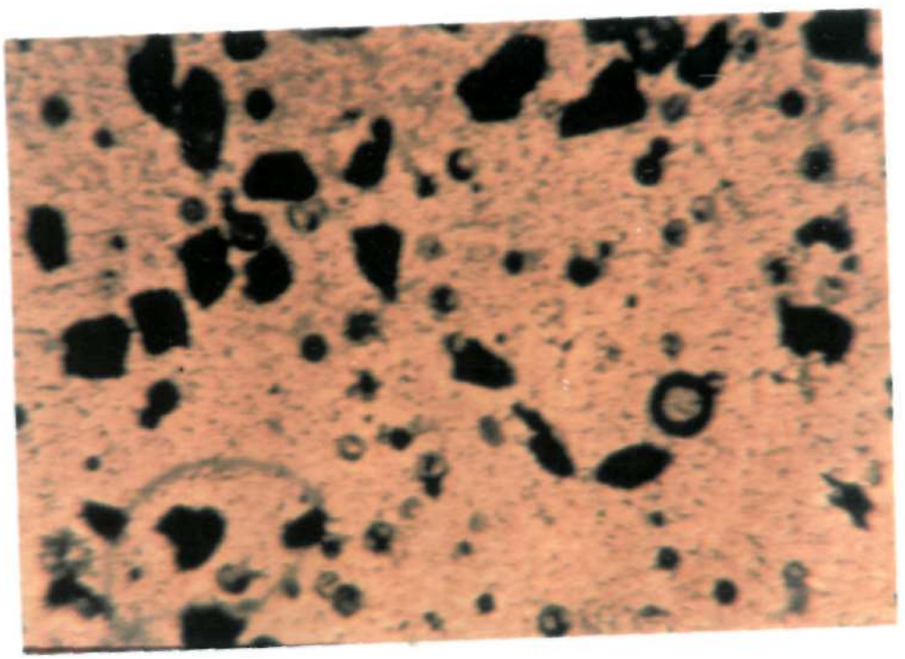
At the north central portion appears altered plagioclase feldspar.

The sub-hedral grains with cracked fractures reveal pyrite with its distinctive cleavages and chalcopyrite.

The yellow inclusion in the sulphide phase is possibly gold.

Photomicrograph of the -300 + 150μm Particle Size Fraction of Desbarats Alluvial Gold ore under Plane Polarized Light. (Magnification = 40)

PLATE VII



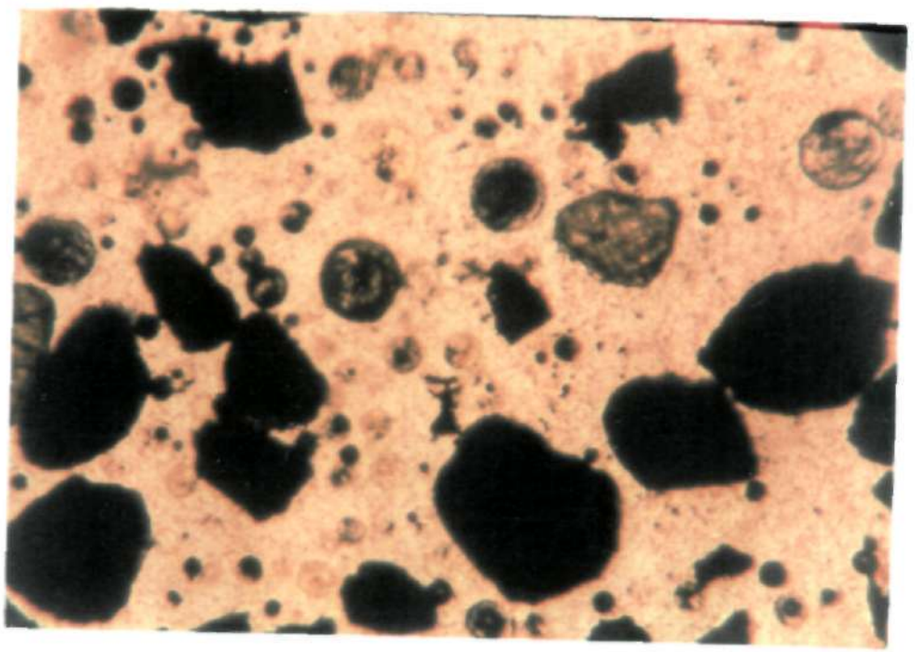
Photomicrograph of the -75μm Particle Size Fraction of Iesha Alluvial Gold ore under Plane Polarized Light. (Magnification - 40)

Ilmenite is the majority of the minerals in the opaque grains while quartz dominates the transparent portion.

Round spots are air bubbles.

The sulphide phase is not easily discernible.

PLATE VI



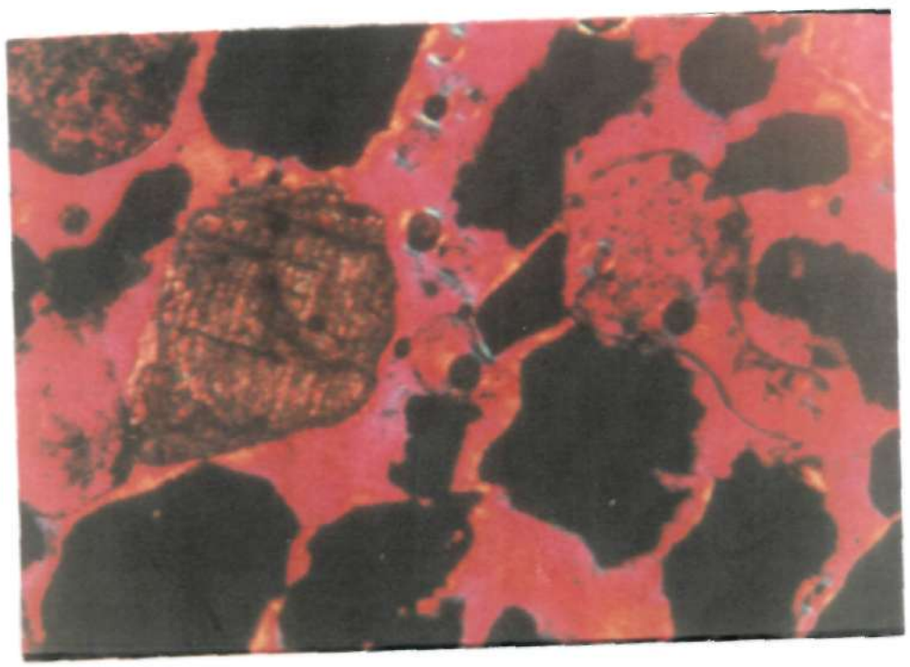
Photomicrograph of the -150 +75μm Particle Size Fraction of Ilesha Alluvial Gold ore under Plane Polarized Light. (Magnification - 40)

The opaque mineral grains is essentially ilmenite.

The brownish yellow portion is pyrite and chalcopyrite.

The rest is quartz and feldspar.

PLATE VIII

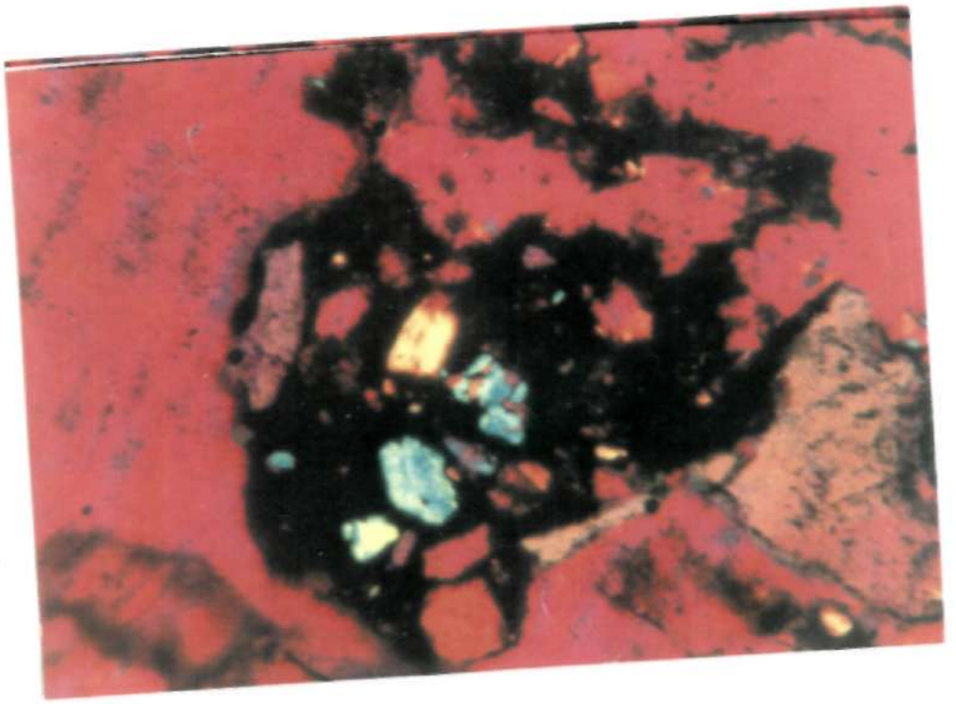


Photomicrograph of the unsized concentrate (-20000μm) of Ilesha Alluvial Gold ore under Cross Nicols. (Magnification - 40)

The relative abundance and mineralogical composition of this size fraction is same as the sample in Plate I.

Here the distintive cleavage of pyrite is clearly revealed.

PLATE IX

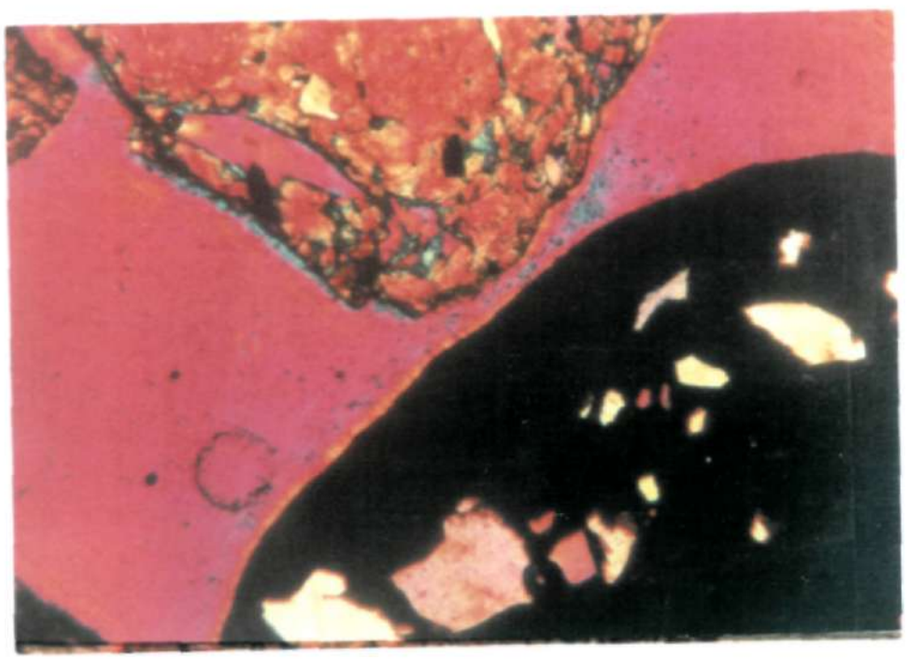


Photomicrograph of the -2000 +850μm Particle Size Fraction of Ilesha Alluvial Gold ore under Cross Nicols. (Magnification × 40)

The quartz/feldspar inclusion in the iron matrix shown in Plate II are clearly shown under cross polars.

The green visible twinning and cleavage of the feldspar grains can be seen while the unaltered yellow crystals of quartz is easily discernible.

PLATE X



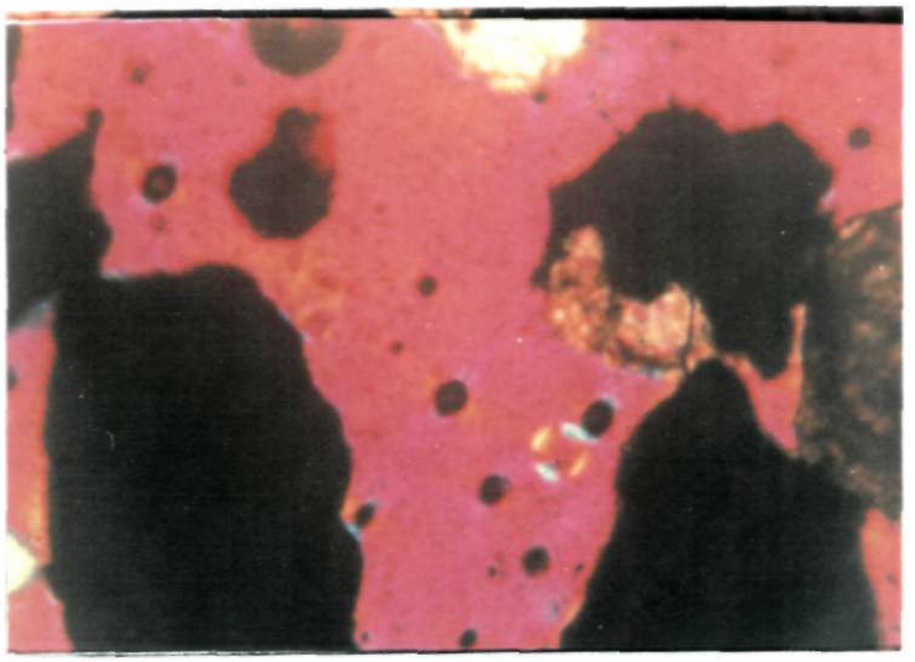
Photomicrograph of the -850 +600μm Particle Size Fraction of Ilesha Alluvial Gold ore under Cross Nicols. (Magnification × 40)

The quartz and feldspar inclusions in the opaque portion of the matrix are clearly revealed.

The subangular brownish grains are the feldspar while the yellow portion is the quartz.

The green coloured chalcopyrite grains in the northern portion are clearly revealed.

PLATE XI

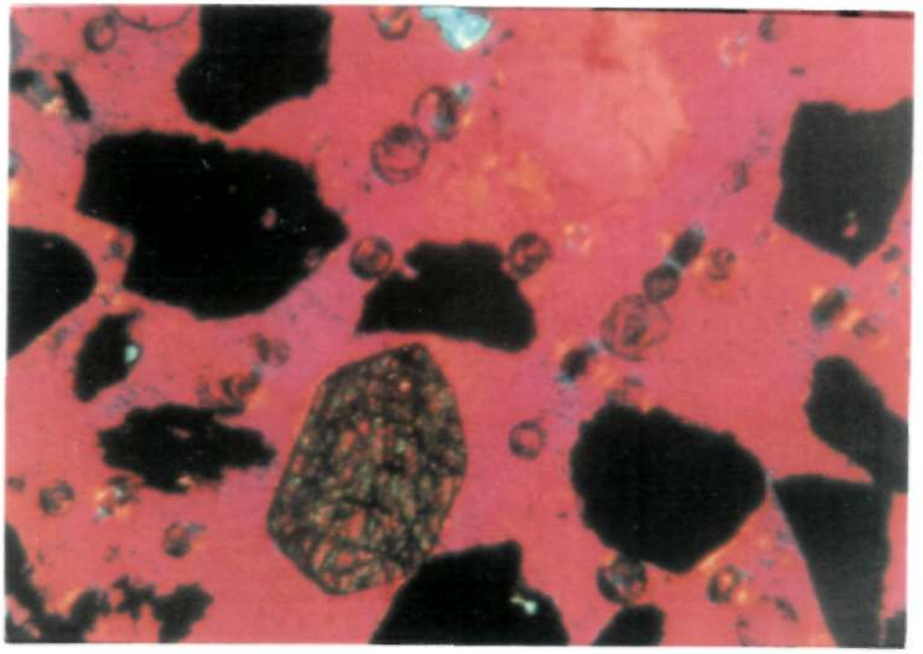


Photomicrograph of the -600 +300μm Particle Size Fraction of Ilesha Alluvial Gold ore under Cross Nicols. (Magnification × 40)

The relative abundance and mineralogical composition of this size fraction is the same as the sample in Plate IV.

The big green grain of chalcopyrite is clearly revealed.

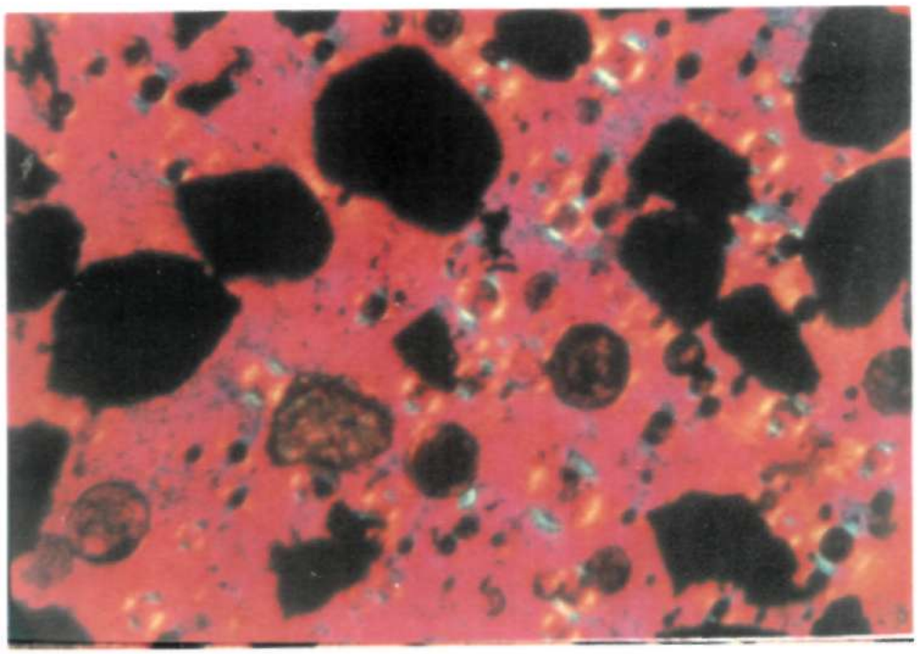
PLATE XII



Photomicrograph of the -300 +150μm Particle Size Fraction of Ilesha Alluvial Gold ore under Cross Nicols. (Magnification × 40)

The relative abundance and mineralogical composition of this size fraction is the same as the sample shown in Plate V.

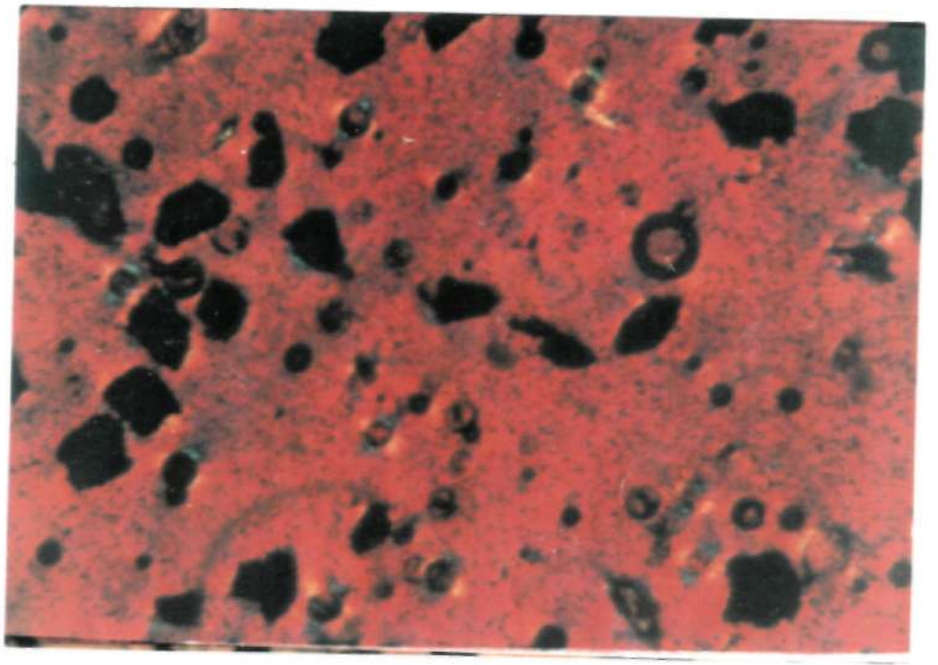
PLATE XIII



Photomicrograph of the -150 +75μm Particle Size Fraction of Lesho Alluvial Gold ore under Cross Nicols. (Magnification × 40)

The relative abundance and mineralogical composition of this size fraction is the same as the sample shown in Plate VI.

PLATE XIV



Photomicrograph of the -75μm Particle Size Fraction of Ilesha Alluvial Gold ore under Cross Nicols. (Magnification × 40)

The relative abundance and mineralogical composition of this size fraction is the same as the sample shown in Plate VII.

with etched and abrasion polished surfaces. Ilmenite ($FeO.TiO_2$)is the most common phase. Crystals of sulphide phases especially chalcopyrite ($CuFeS_2$) and pyrite (FeS_2) were also observed. Few grains still retain distinctive cleavages and a wide variety of exsolved inclusions. These inclusions occur as shiny specks some of which are quartz and, possibly gold. Such mode of occurrence for the gold as inclusions rather than free crystals phases is documented in this goldfield (Elueze, 1982; Ajayi,1988; Suh, 1993).

Shown in Table 4.3 are the results of modal analysis of Ilesha gold ore.

Table 4.3 Modal Analysis Showing Relative Proportion Of Mineral Species in the Sample

Slide Number	Particle Size μm	Minerals Percent				
		Pyrite	Chalcopyrite	Ilmenite	Other Opaques	Quartz/Feldspar
1	+2000	10	10	50	10	15
2	-2000 +850	-	-	15	30	55
3	-850 +600	-	12	30	28	30
4	-600 +300	5	15	30	20	30
5	-300 +150	20	15	30	25	10
6	-150 +75	17	18	40	5	20
7	-75	15	10	25	10	40

The opaque grains of the +2000μm, -2000 +850μm, -850 +600μm size ranges are quite remarkable because the opaque cores are rimmed by a reddish-brown ferrous coat. This coat layer carries a lot of quartz inclusions and sulphide specks. From the modal analysis (Table 4.3) the sulphide contents for the +2000μm, -2000 +850μm, -850 +600μm, -600 +300μm, -300 +150μm, -150 +75μm, and -75μm size fractions are approximately 20%, 0%, 12%, 20%, 35%, 35% and 25% respectively. Although no definite trend is deciphered from the table, it appears that the sulphide content increases with decrease in particle size.

4.1.4 X-ray Diffractometry

The diffractograms obtained from x-ray diffractometry of the different particle sizes of the sample are shown in Figures 4.2 - 4.8. The main opaque mineral phase is ilmenite ($FeO.TiO_2$) with minor amount of haematite, geothite,

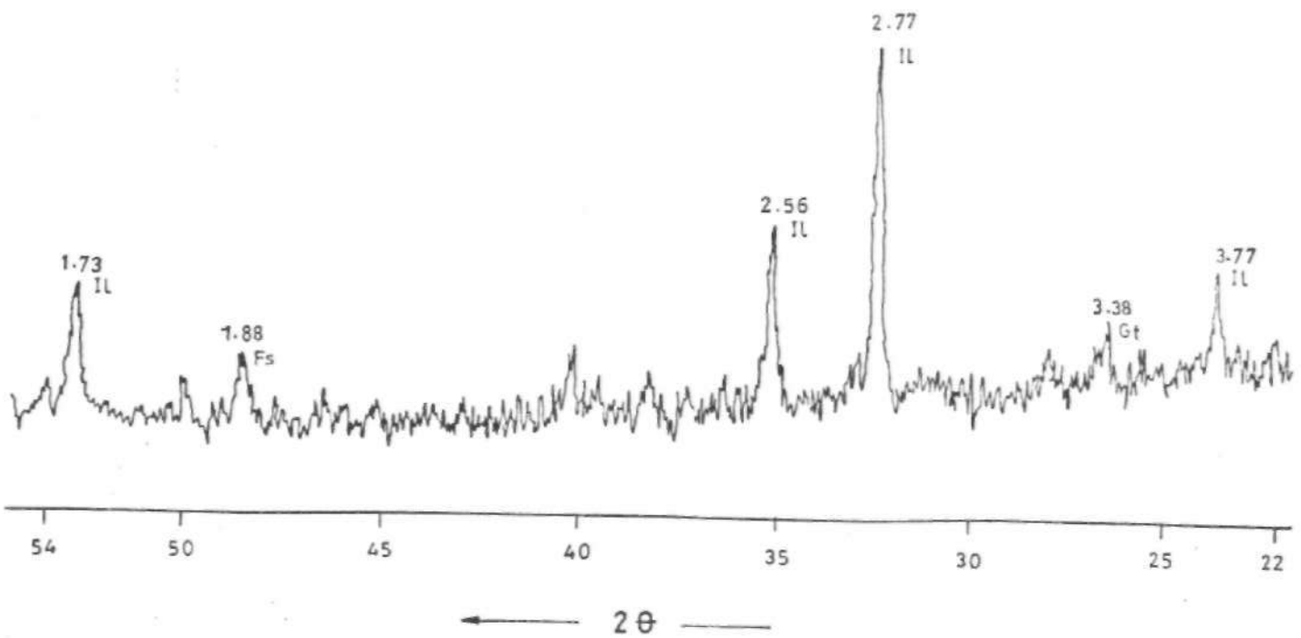


Fig. 4.2 DIFFRACTOGRAM OF THE UNSIZED CONCENTRATE (+2000μm)
(Il - Ilmenite; Gt - Goethite; Fs - Feldspar).

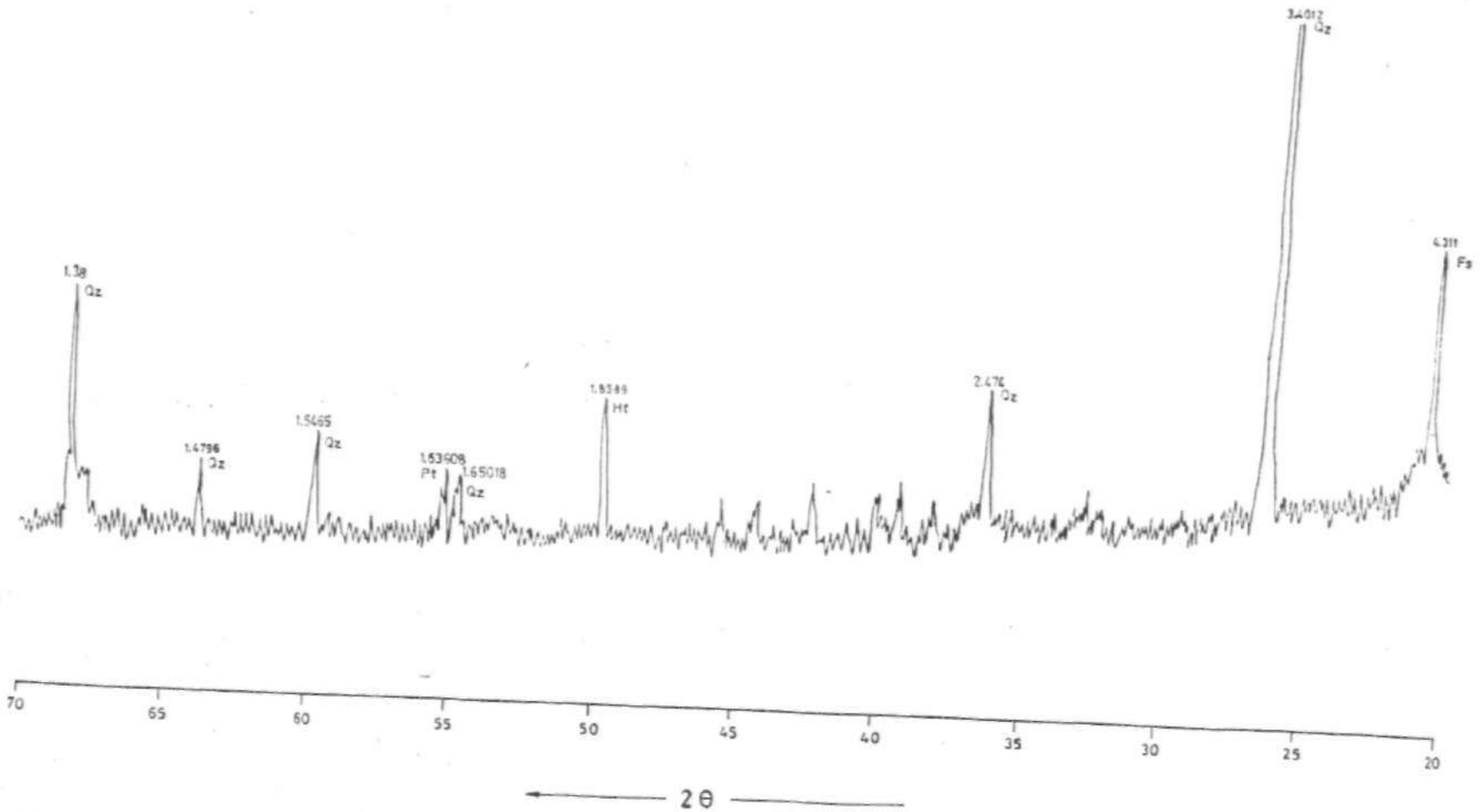


Fig. 4.3 DIFFRACTOGRAM OF −2000 + 850μm PARTICLE SIZE FRACTION
(Fs − Feldspar ; Qz − Quartz; Ht − Hematite ; Pt − Pyrite).

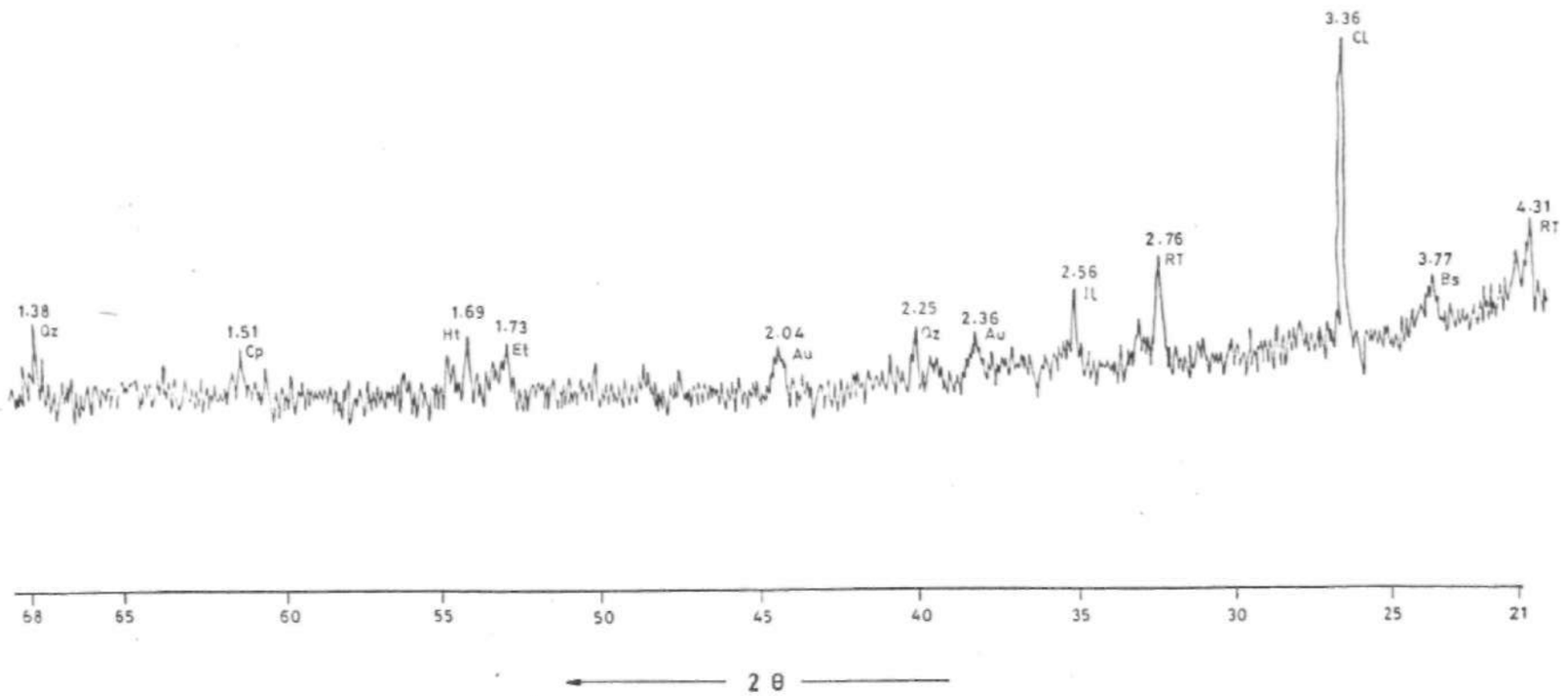


Fig. 4.4 DIFFRACTOGRAM OF -850 +600μm PARTICLE SIZE FRACTION
(RT- Reniete; Bs - Barium sulphate ; Cl - Chrysocola ; Rt - Rathite ; IL - Ilmenite; Au - Gold ; Qz - Quartz ; Et - Ellestadite ; Ht - Hematite ; Cp - Chalcopyrite).

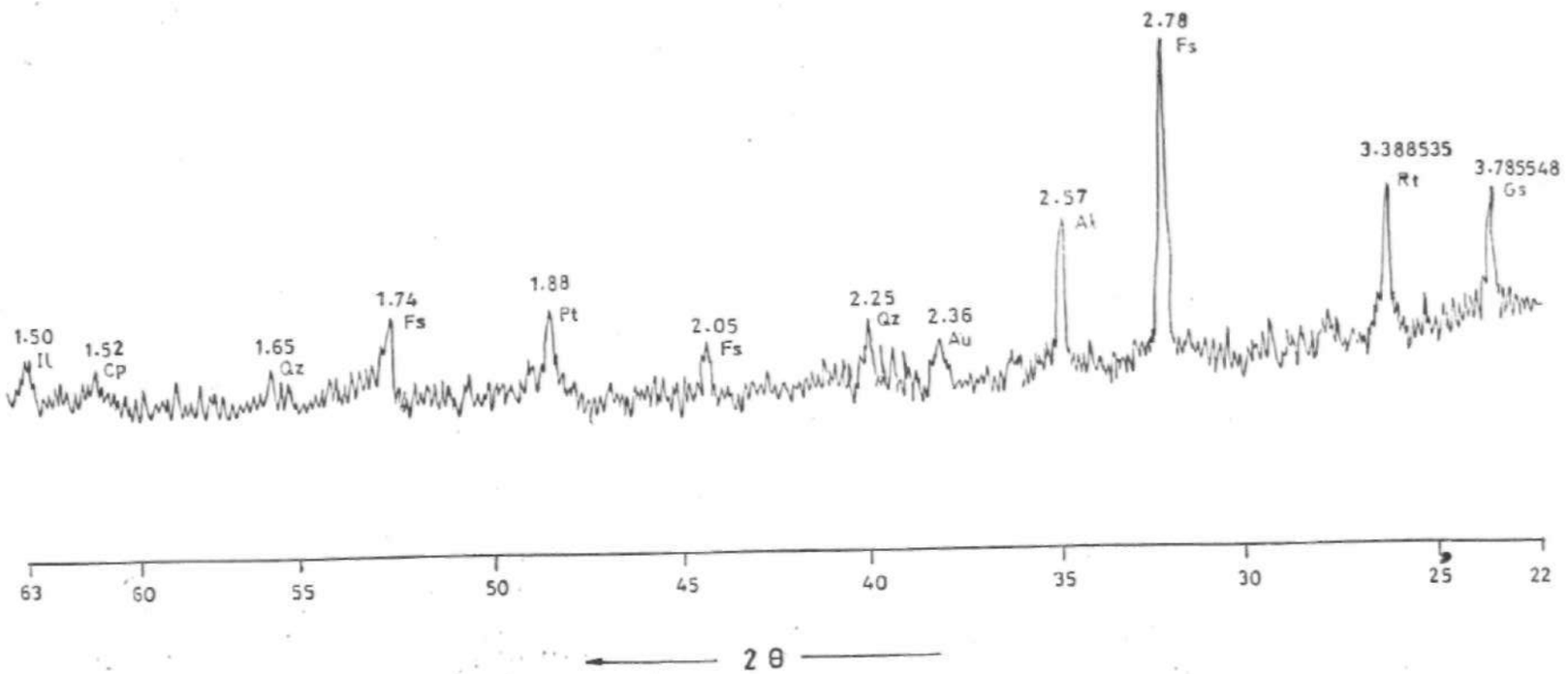


Fig. 4.5 DIFFRACTOGRAM OF -600 + 300μm PARTICLE SIZE FRACTION
(Gs - Gypsum ; Rt - Rathite ; Fs - Feldspar ; Al - Almandine ; Au - Gold ; Qz - Quartz ; Pt - Pyrite ; Cp - Chalcopyrite).

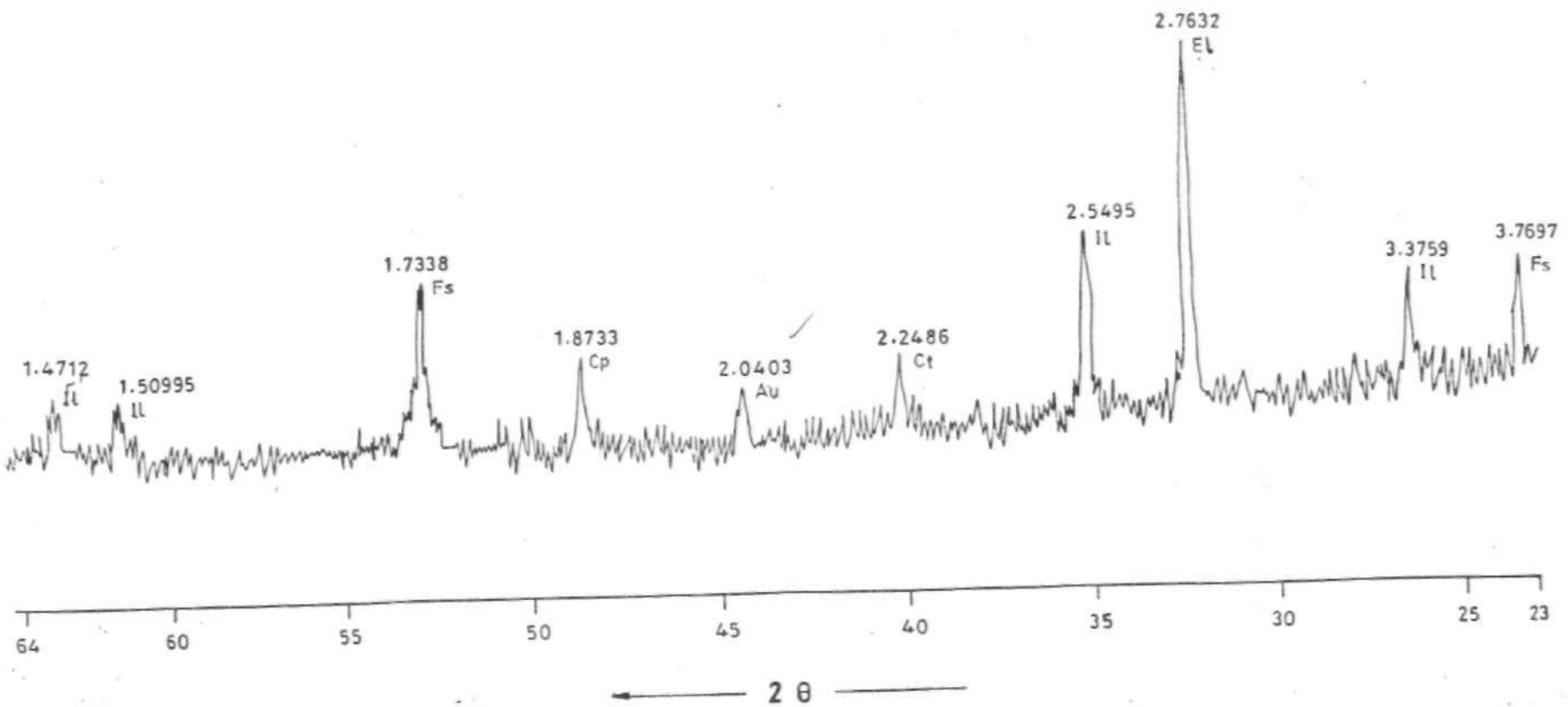


Fig. 4.6 DIFFRACTOGRAM OF -300+150μm PARTICLE SIZE FRACTION
(Fs-Feldspar ; Il – Ilmenite ; El-Ellestadite; Ct-Cattierite ; Au-Gold ; Cp-Chalcopyrite).

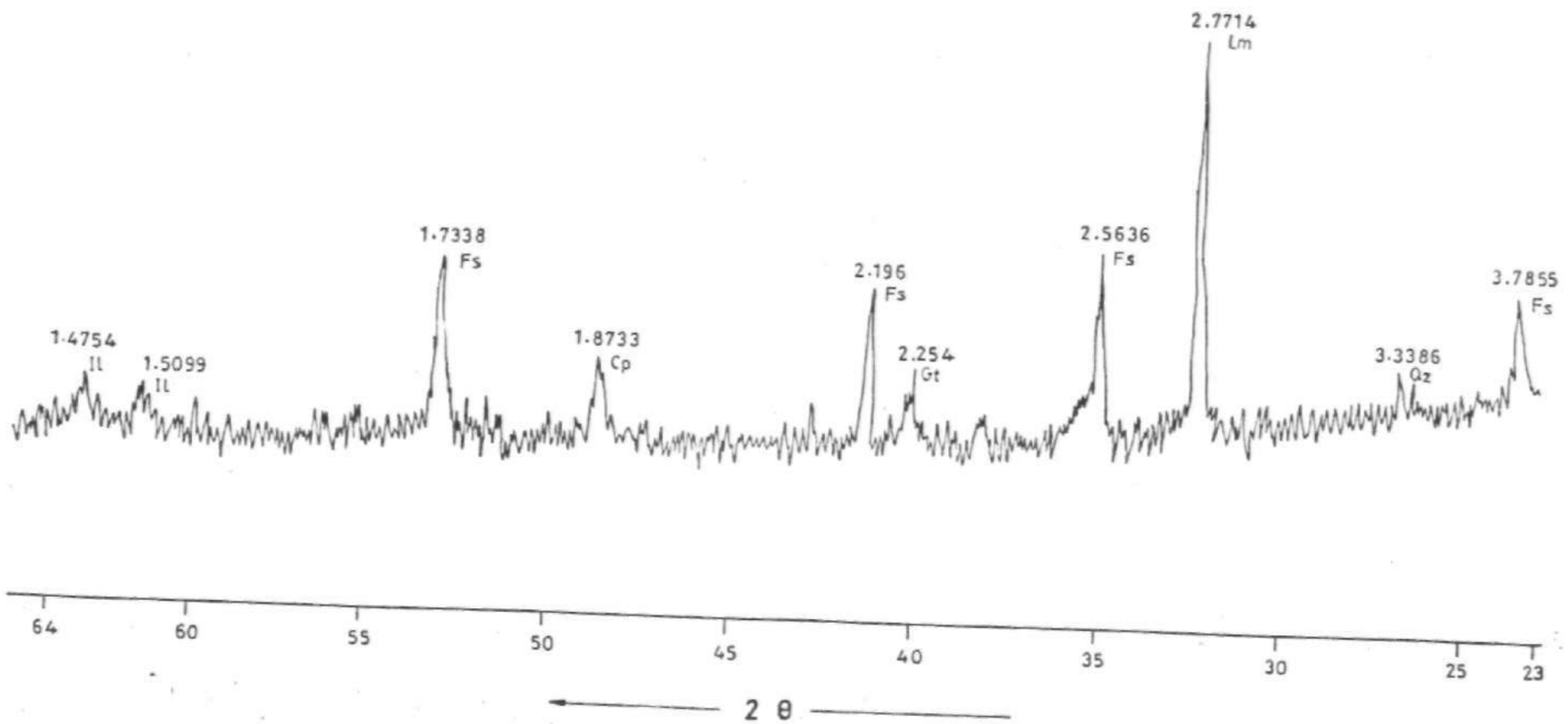


Fig. 4.7. DIFFRACTOGRAM OF -150 +75μm PARTICLE SIZE FRACTION
(Il- Ilmenite; Fs- Feldspar; Cp- Chalcopyrite; Gt- Geothite; Lm-Lime; Qz-Quartz).

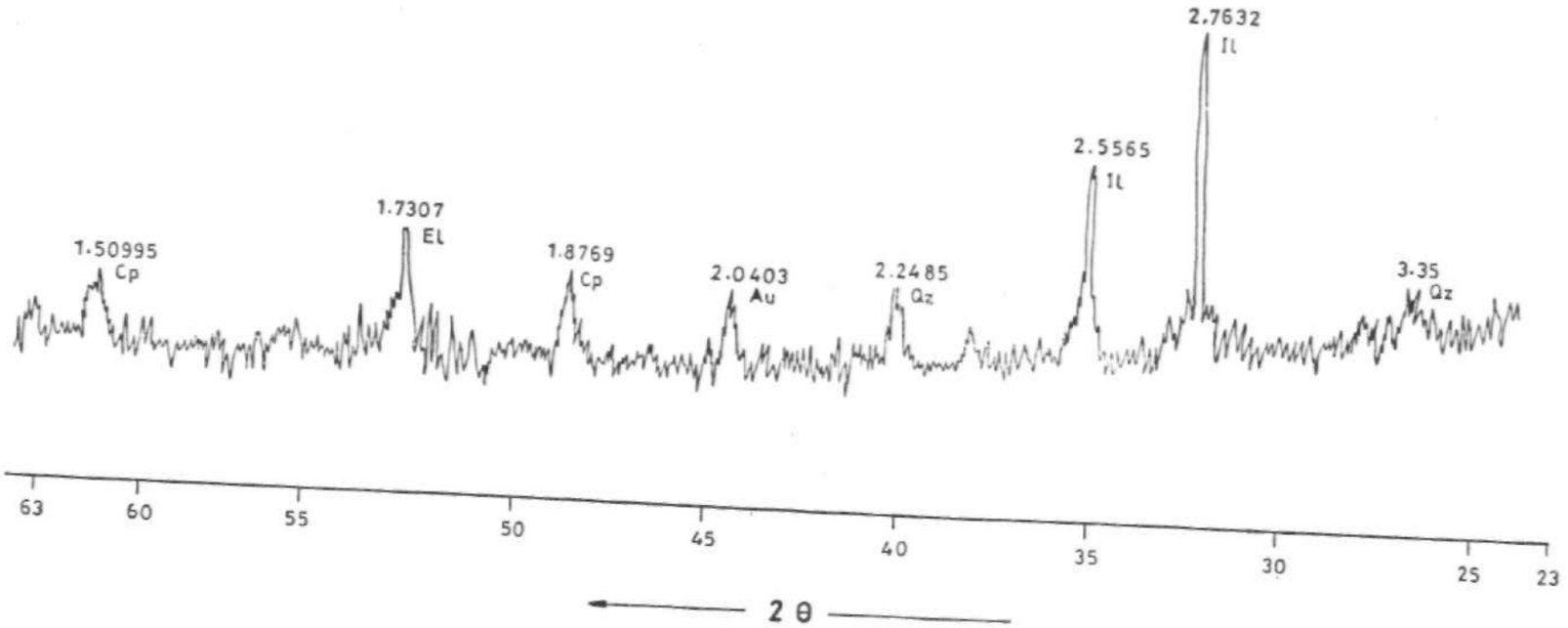


Fig. 4.8 DIFFRACTOGRAM OF -75 μm PARTICLE SIZE FRACTION
(Qz-Quartz; Il-Ilmenite; Au-Gold; Cp - Chaleopyrite; El - Ellestadite).

and/or quartz and subordinate amount of feldspar. Pyrite and chalcopyrite also show prominent peaks. Although, free gold is hardly discernible on the microscope, it is significant to note that a distinct gold peak occurs in each of the diffractograms shown in Figure 4.4, 4.5, 4.6 and 4.8. The chemical formulae of the various minerals in the diffractograms are contained in appendix 2.

4.2 Results Of Ore Analysis

The results of analysis to determine the gold content in the ore samples and other associated metals are contained in this section.

4.2.1 Fire-Assay-Gravimetric Analysis

Table 4.4 presents the result of gold content determination by fire-assay-gravimetric method. The assay value of +2000μm size fraction is 9.2 g/ton of gold while that of -75μm is 152.8 g/ton of gold (the highest value). Generally, the assay value of the gold increases with decrease in particle size.

Table 4.4 Gravimetric Fire-Assay Test Results

Sample Number	Size Fraction (μm)	Assay Value (g/ton) of Au
1	+2000	9.2
2	-2000 +850	12.4
3	-850 +600	9.6
4	-600 +300	20.0
5	-300 +150	25.0
6	-150 +75	26.2
7	-75	152.8

4.2.2 Atomic Absorption Spectrophotometry

For confirmation of results in Table 4.4, analysis of Igun gold ore samples were subjected to atomic absorption spectrophotometric (AAS) analysis and result obtained is presented in Table 4.5.

Table 4.5 Analysis of Gold by AAS

Sample No.	Volume Used (mls)	Particle Size (μm)	Absorbance $\times 10^{-3}$ 1	2	3	4	Average Absorbance	Gold Content (ppm)
1	50	+2000	24	23	-	-	0.0235	4.666
2	50	-2000 +850	13	16	13	17	0.015	2.9453
3	50	-850 +600	7	5	6	-	0.006	1.1235
4	50	-600 +300	7	5	5	-	0.0053	0.9818
5	50	-300 +150	30	35	34	-	0.033	6.5891
6	50	-150 +75	68	66	-	-	0.067	13.4717
7	50	-75	42(×10)	47(×10)	-	-	0.0445(×10)	89.11

The unsized concentrate (+2000μm) size fraction has a gold content of 4.666ppm and while the least size fraction (-75μm) has a gold content of 89.11ppm (the highest value). From +300μm downwards, it is observed that gold content increases with decrease in particle size.

4.2.3 Test for Significance Difference Between AAS and Fire-Assay-Gravimetric Readings

Statistical analysis using student t-test with the usual notation:

$$t = \frac{\overline{x}_1 - \overline{x}_2}{s\sqrt{\frac{1}{n_1} + \frac{1}{n_2}}}$$

where

$$s^2 = \frac{\sum_1 (x - \overline{x}_1)^2 + \sum_2 (x - \overline{x}_2)^2}{n_1 + n_2 - 2}$$

	Fire-Assay values x_1	$(x_1 - \overline{x}_1)^2$	AAS values x_2	$(x_2 - \overline{x}_2)^2$
	9.2	743.1076	4.666	151.3459
	12.4	578.8836	2.9453	196.9728
	9.6	521.4596	1.1235	251.4286
	20.0	270.9316	0.9818	255.9424
	25.0	131.3316	6.5891	107.9708
	26.2	105.2676	13.4717	12.3082
	152.8	13534.995	89.1100	5202.7369
n	7	7	7	7
$\overline{x}$	36.46	2297.9966	16.98	882.7135
$\sum x$	255.2	15885.976	118.89	6178.9942

$$
\begin{aligned}
s^2 &= \frac{15885.976 + 6178.9942}{7+7-2} \\
&= \frac{22064.97}{12} \\
&= 1838.7475 \\
t &= \frac{36.46 - 16.98}{42.8806\sqrt{\frac{1}{7}+\frac{1}{7}}} \\
&= \frac{19.48}{22.9206} = 0.84988 \\
t &= 0.84988 \text{ at } 12df.\ p > 0.05
\end{aligned}
$$

The 2.179 value of t at $n_1 + n_2 - 2 = 12df$ and $P = 0.05$ exceeded the calculated value (0.84988). We therefore accept Null hypothesis and conclude that there is no significant difference between the Au readings from Fire-Assay and AAS procedures.

4.2.4 AAS Analysis for Silver, Copper, Lead and Zinc

The results of atomic absorption spectrophotometric analysis for silver, copper, lead and zinc content in the Igun gold ore samples are presented in Tables 4.6 and 4.7.

Table 4.6 Analysis of Silver, Copper and Lead by AAS

Sample No	Size Fraction (μm)	Absorbance $\times 10^{-3}$ Ag	Cu	Pb	Sensitivity's S=0.0044×2 /Absorbance
1	+2000	< DL	< DL	< DL	
2	-2000 +850	< DL	< DL	< DL	
3	-850 +600	< DL	< DL	< DL	
4	-600 +300	< DL	< DL	< DL	
5	-300 +150	< DL	< DL	< DL	
6	-150 +75	< DL	< DL	< DL	
7	-75	< DL	< DL	< DL	

< DL ⇒ metal content is below the detection level of AAS.

Table 4.7 Analysis of Zinc by AAS

Sample No.	Size Fraction (μm)	Absorbance $\times 10^{-3}$		Average Absorbance	ppm of Zn
1	+2000	89	98	0.0935	0.9905
2	-2000 +850	102	94	0.098	1.0333
3	-850 +600	103	96	0.0995	1.0475
4	-600 +300	95	98	0.0965	1.022
5	-300 +150	123	119	0.121	1.2518
6	-150 +75	128	125	0.1265	1.304
7	-75	146	148	0.147	1.4988

It is observed from the Tables that while silver, copper and lead cannot be traced in the samples, zinc content ranges from 0.9905ppm in the +2,000μm size fraction to 1.4988ppm in the -75μm size fraction.

4.3 Experimental Results

4.3.1 Amalgamation

The results of amalgamation tests on Ilesha gold ore are shown in Table 4.8

Table 4.8 Results of Amalgamation Tests on Ilesha Gold Concentrate

Size Fraction (μ)m	Weight of Crucible & Sample (g)	Weight of Sample (g)	Weight of Crucible + Au values after retorting	Weight of Au values (g)$\times 10^{-3}$	Gold Recovered g/ton	Fire Assay g/ton Au	% Recovery
+2000	16.88000	5.00000	11.88002	0.02	4.00	9.20	43.48
-2000 +850	20.90000	10.00000	10.90000	0.00	0.00	12.40	0.00
-850 +600	21.44000	7.50000	13.94001	0.01	1.33	9.60	13.85
-600 +300	31.87000	20.00000	11.87001	0.01	0.50	20.00	2.50
-300 +150	32.24000	20.00000	12.24000	0.01	0.50	25.00	2.00
-150 +75	18.10000	2.50000	15.60000	0.00	0.00	26.90	0.00
-75	12.03000	0.17000	11.86002	0.02	58.82	152.80	38.49

There is no discernible trend observed in the recovery of gold by amalgamation in the various size fractions. One thing is obvious: The recovery is generally low. Size fractions -2000 +850μm and -150 +75μm have zero recoveries.

4.3.2 Percolation Leaching

In the first experiment (gold recovery versus cyanide concentration) as shown in Figure 4.9, it is observed that recovery increases with cyanide concentration.

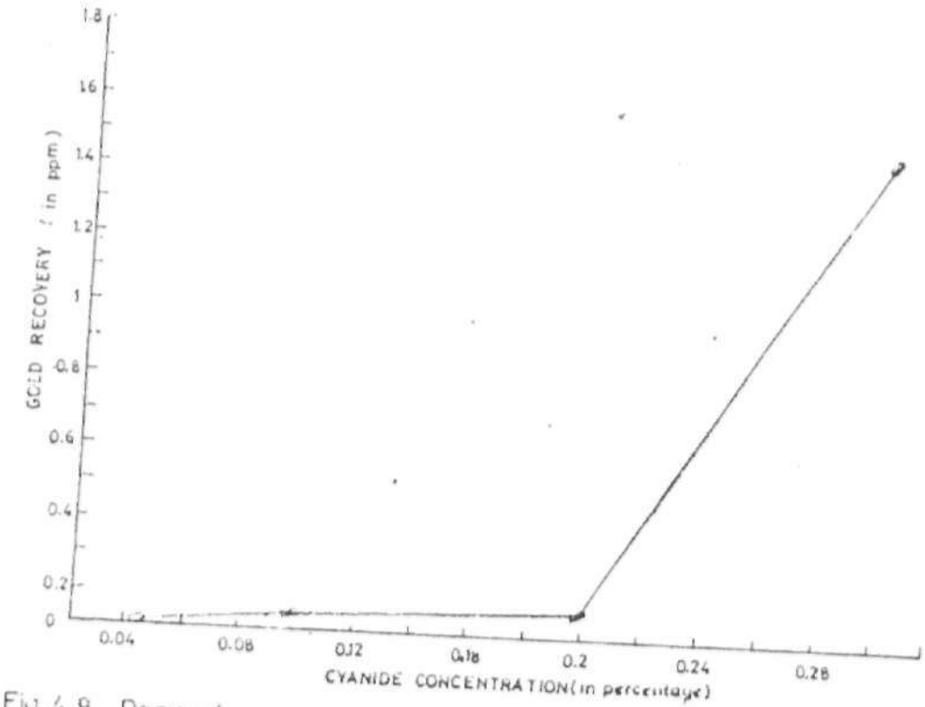


Fig. 4.9 Dependence of gold recovery on cynide concentration.

For cyanide concentration of 0.05%, 0.0265ppm gold was recovered by percolation leaching (under the experimental conditions of pH = 11.5, retention time of 4 hours, for unsized concentrate). For 0.3% cyanide concentration, 1.4429ppm of gold (the highest value) was recovered. Thus 0.3% cyanide was used in the next experiment in order to optimise gold recovery.

The results of gold recovery versus retention time are shown in Table 4.9. It is observed that gold recovery increases with retention time up to the retention time of six-hours, thereafter the gold recovery begins to drop.

The results of gold recovery versus hydrogen ion concentration are shown in Figure 4.10.

Here the pH of 11.5 gives the recovery of 4.4365 ppm (the highest) and so was used for the next experiment.

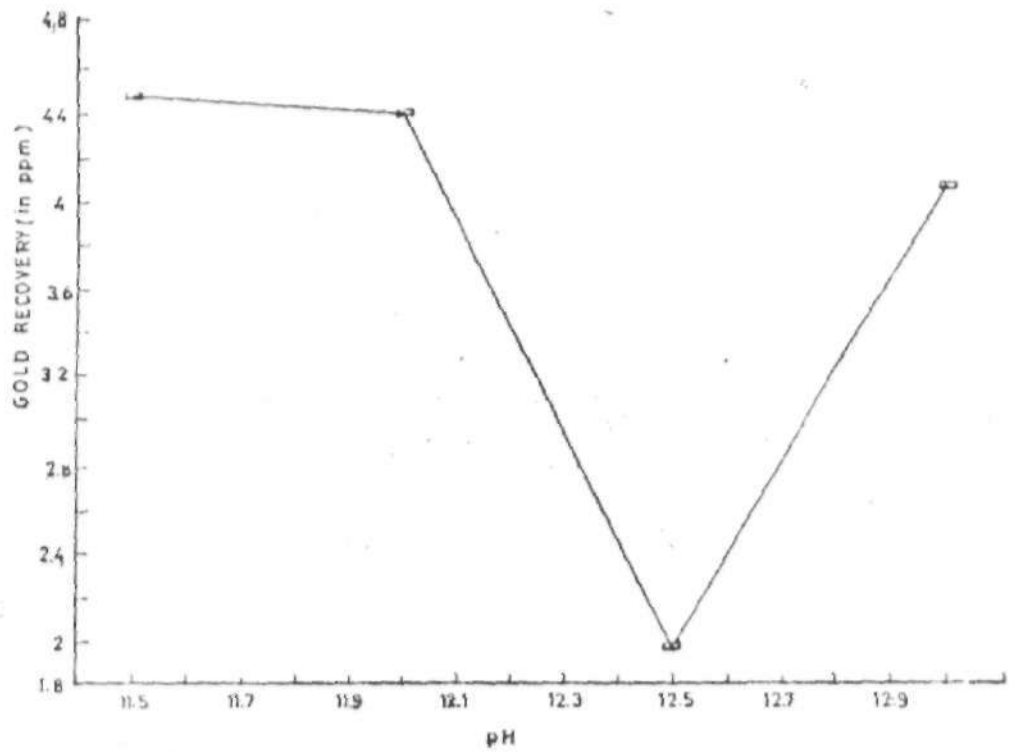
The results of gold recovery versus particle size are shown in Figure 4.11.

Table 4.9: Gold Recovery as Function of Retention Time

Retention Time (hour)	Absorbance $\times 10^{-3}$			Average Absorbance	Gold Recovery (ppm)
	1	2	3		
4	24	24	-	0.024	1.1128
6	131	133	126	0.130	4.5973
7	87	88	85	0.087	3.1828

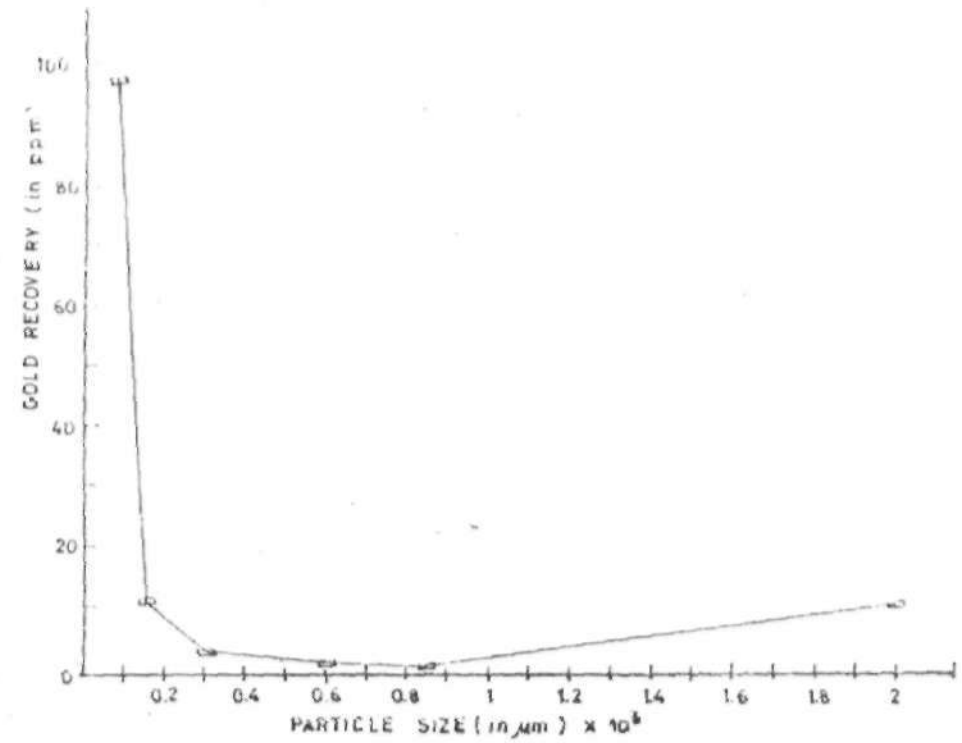
It is observed that on applying the optimum leaching parameters (of 0.3% CN^-, pH of 11.5 and 6 hour retention time) to the smallest particle size fraction of -75μm the highest gold recovery of 97.185ppm (97.185g/ton) was obtained.

Percentage recoveries of gold in the various size fractions by percolation leaching are shown in Table 4.10



4.10

Fig 4.10 Effect of pH on gold recovery.



4.11

Fig.4.11 Particle size effect on gold recovery.

Table 4.10 Percentage Recoveries of Gold in the Various Size Fractions by Percolation Leaching.

Particle Size fraction(μm)	Gold Content by Fire-Assay (g/ton)	Gold Recovered in ppm (g/ton)	% Recovery
Unsized concentrate	09.20	-	-
-2000 +850	12.40	0.6375	5.14
-850 +600	09.60	4.4879	46.75
-600 +300	20.00	1.6339	8.17
-300 +150	25.00	2.7097	10.84
-150 +75	26.90	10.8499	40.33
-75	152.80	97.1850	63.60

4.4 Discussion of Results

4.4.1 Mineralogical Characterization of Igun Gold Ore

The result of the screen analysis shown in Table 4.2 is quite revealing. -600 +300μm size fraction alone carries 57.50% of the gold values in the ore sample. Thus the bulk of the gold values are contained in the finer size fractions.

The result of petrographic studies of Igun gold ore is shown in Table 4.3. The sulphide phase constitute 20%, 0%, 12%, 20%, 35%, 35%, 25% for +2000μm, -2000 +850μm, -850 +600μm, -600 +300μm, -300 +150μm, -150 +75μm and -75μm size fractions respectively. +2000μm is unsized concentrate. Baring the anomalous cases of -2000 +850μm and -75μm size fractions, the general trend therefore indicates that the gold content increases with the amount of sulphide phase in the particle size fraction. Evidently, the sulphide phase accomodates good proportion of the gold values. This result corroborates the work earlier on carried out on Waya Gold field (N.W. Nigeria) which is part of the Pan-African mobile belt (Woakes, M. and Bafor, B.E. 1984) as Ilesha gold field (W. Nigeria) (Garuba, 1988). For the particle size of -2000 +850μm ilmenite is only 15% but other unidentifiable opaque minerals constitute as high as 30% of the sample mineral content. This peculiar size fraction has no discernible sulphide phase on the microscope neither was free gold detected by x-ray diffractometry. There is the possibility of gold occurring in the opaque portion of this size fraction as nickelliferrous pyrrotite as being documented in Ilesha/Ife gold field (Suh, 1993). The assay value (by fire-assay-gravimetric method) of the -75μm size fraction is 152.8g/ton which is very high when compared with the average gold content of the bulk sample which is 19.143g/ton. With lower sulphide content why do we have high assay value in the -75μm fraction? The plausible explanation is that there is free gold in this size fraction. The apparent puzzle is that no free

gold is discernible on the microscope. Rather the quartz/feldspar constituent of this size fraction is high (40%). It is an established fact that quartz being very stable has its content increases with decrease in particle size. At this very fine size fraction (-75μm) the sulphide crystals are too small for easy identification on the ore microscope and better results (to identify more sulphide phases) should be obtained by scanning electron microscope.

It is quite significant to observe from the x-ray diffractograms shown in Figures 4.4, 4.5, 4.6 and 4.8 that a distinct gold peak occurs in each of the following size fractions: -850 +600μm, -600 +300μm, -300 +150μm and -75μm. The high assay value of the -75μm size fraction can only be explained by the fact that apart from the finely disseminated gold locked up in the sulphide phase, free gold does occur to some extent. This reinforces the postulation that Ilesha gold ore belongs to the non-refractory deposit. However, the alluvial deposit is finely grained and largely sub-microscopic gold. This understanding is useful in the design of recovery/processing programme of Igun gold.

Although, from +2,000μm to 600μm the results of gold assay values through AAS and fire-assay gravimetric methods (Tables 4.4 and 4.5) do not follow a discernible trend, it is observed in both cases that from the particle size range of +300μm to -75μm the gold assay value increases with decrease in particle size with the -75μm having the highest gold content (of 89.170g/ton and 152.8g/ton respectively). Statistical analysis of the results of fire-assay-gravimetric and AAS ore analysis show that there is no significant difference between the two techniques used to determine gold content. Thus it is valid to conclude that the finer the particle size, the richer the gold content. The ore analysis shows that Ag, Pb, Cu, are not associated with Igun gold deposit but Zn does. The form of occurrence and the effects on gold recovery efficiencies are yet to be investigated.

4.4.2 Extractive Metallurgy of Igun Gold

It was reported that gold was won by passing the liberated ore over gold plate coated with mercury. It extracted the precious metal with an efficiency of between 75 and 80 percent. When pyritic ore was encountered, the recovery fell below 50 percent (Cowey, 1994). Thus it is not much of a surprise that the recovery of Igun gold by amalgamation is low since most of the gold appeared to be locked up in the sulphide phases. The size fractions of Igun gold ore that have free gold in them are somewhat amenable to amalgamation method of recovery. The qualified fractions as observed from the x-ray diffractogrammes are -850 +600μm, -600 +300μm, -300 +150μm and -75μm. Amalgamation is typically a subsidiary process combined with flotation and cyanidation (Pearl, 1973). Percolation leaching is considered for the processing of Igun gold ore because this method is applicable to low-grade ore; moreso, when used with screen analysis which ensures regularity in the size of the particle.

Expectedly, the recovery of gold increases with increase in cyanide concentration as shown in Figure 4.9 (Table 4.16 in Appendix 10). Gold recovery increases with retention time until 6 hours retention time is attained; thereafter recovery decreases. A possible explanation is that the decomposition of the CN^- solution with time depending on gold associated minerals may bring about dissolution of the aurocyanide complex. Iron and zinc minerals that are associated with gold dissolve in cyanide and therefore deplete the solution of its cyanide content. Shown below is a possible reaction between zinc metal and free cyanide: $ZnS + 4CN^- \longrightarrow Zn(CN)_4^{2-} + S^{2-}$.

On the other hand, increasing the pH of the solution decreases the gold dissolution and hence the recovery as observed in Figure 4.10 (Table 4.18 in Appendix 10). That notwithstanding, it is essential that the solution be kept alkaline to minimize hydrolysis of cyanide ion: $CN^- + H_2O \longrightarrow HCN + OH^-$ and to minimize the absorption of CO_2 which is capable of decomposing

cyanide ion: $CN^- + H_2CO_3 \longrightarrow HCN + HCO_3^-$ (Habashi, 1970). HCN is so toxic that its formation should by all means be prevented. In aqueos solutions, cyanide ion hydrolysis to form hydrogen cyanide (HCN) with a Pka = 9.21 at 25^0C (Smith and Martell, 1976). It is a difficult task to predict the behaviour of the associated gangue minerals. Components like Cu, Fe and Zn are troublesome as they greatly increase the consumption of both cyanide and oxygen (Hedley and Tabachnick, 1958). Calcium ion has no effect on gold dissolution (Habashi, 1970). However, at pH > 11.5 it has a retarding effect which is presumably due to the formation of calcium peroxide on the metal surface and this prevents reaction with cyanide. Calcium peroxide was considered to be formed by the reaction of lime with H_2O_2 accumulating in the solution according to: $Ca(OH)_2 + H_2O_2 \longrightarrow CaO_2 + 2H_2O$.

The fact that gold recovery increases with decrease in size of the particle is not only because of the large surface area per unit weight exposed to the attack of the complex cyanide ions but also because of the presence of some free gold in the fine particles as earlier on observed. Gold dissolution is a function of characteristics of gold and detrimental coatings (such as iron oxide) on the gold particles.

It has been postulated that Ilesha alluvial gold ore is essentially a non-refactory or readily leached ore. The experimental results on this gold ore show that it is amenable to percolation leaching. The gold recovery in the particle size fractions of -2000 +850μm, -850 +600μm, -600 +300μm, -300 +150μm, -150 +75μm and -75μm are 0.6375 ppm, 4.4879 ppm, 1.6339 ppm, 2.7097 ppm, 10.8499 ppm and 97.185ppm respectively. When compared with the fire assay values indicated in Table 4.4, the percentage recoveries (when the optimum conditions of pH of 11.5, CN^- concentration of 0.3% and retention time of 6 hours are applied to six particle size fractions) are 6.93%, 36.19%, 17.07%, 13.55%, 41.41% and 63.60% respectively are obtained (Table 4.10).

It has been observed that even though the bulk of the gold is in the -600 +300μm size fraction, (57.06% by weight) the recovery in this size fraction is as low as 17.07%. We have to go down to the particle size fraction of -75μm to obtain a recovery of 63.60% (Table 4.10). If the process parameters are adequately optimised, cyanidation should enhance recovery efficiency of Igun gold ore.

4.5 Flowsheet Development

Recovery of gold from gold ore entails three major sequences of operation:

(i) Mineralogical appraisal of the ore which entails identification of the mineral species, ascertaining the properties of the ore and sizes at which the values may be liberated from the gangues and from one another. Also, it entails exploitation of the different properties that are going to be used in their separation, and ascertaining the size distribution of the ore.

(ii) Appropriate process design: The result of mineralogical appraisal, ore analysis and experimentation lead to the appropriate process design. Laboratory quantity of the ore material was used during this investigation.

(iii) Process evaluation: Having got all the relevant data on process design, the next stage is process evaluation. This entails estimation of the capital and operation cost and comparison of same with the revenue. The process selected for the ore must be appropriate to the magnitude and type of separation criterium. As for the equipment, it is important to know whether or not the through-put is low. The process must be appropriate to the sizes of the mineral and local conditions.

The above criteria form the bases for the proposed process flowchart for Ilesha gold ore.

4.5.1 Flowsheet Development For Igun Gold Ore Based On Experimentation

The leaching method adopted for processing an ore will depend mainly upon its metal content, the cost of mining and bringing the ore to the surface, the cost of milling and any other preleaching treatment such as roasting and

the ease of dissolution. Where the valuable mineral in an ore is finely disseminated, prefential dissolution of the valuable mineral may prove difficult unless the ore is broken to a small particle size (which attracts additional costs), altough liberation is usually unnessary.

From the economic viewpoint, the higher the grade of an ore, the greater is the value tied up in the given amount of the material. In order to minimize interest charges a rapid rate of processing is desirable, but this usually involves higher capital and operating costs. This may be quite acceptable if large resrve are involved to spread the costs. Otherwise it is often a compromise between the rate of processing and total processing costs that provide the optimum. This is the basis for choosing percolation leaching which is discussed in Section 2.3.

A summary of the methods of leaching with their characteristics is given in Table 4.11.

Table 4.11 Methods of Leaching (Jackson, 1986).

Leaching Method	Ore Size	Characteristic Extraction Rate	Type of Cost
Agitation leaching (including pressure leaching)	-30 mesh ($< 0.5mm$)	90 -95 % in 24 hours	High Capital and operating cost
Percolation or Vat leaching	$< 10mm$	$\sim$ 80% in one week	High Capital cost
Heap Leaching	Broken	$\sim$ 50% in Several months	Low Capital and
Dump leaching	as mined	$\sim$ 50% in	operating
In -situ leaching		1 or 2 years	costs

The flowsheet based on the result of amenability assessment of Igun gold to amalgamation and percolation leaching is shown in Figure 4.12. No further consideration was given to amalgamation method of extraction because of the poor response of Igun alluvial gold ore to the method. In the study carried out by Adetunji (1991) on leaching of gold from Ilesha gold ore using

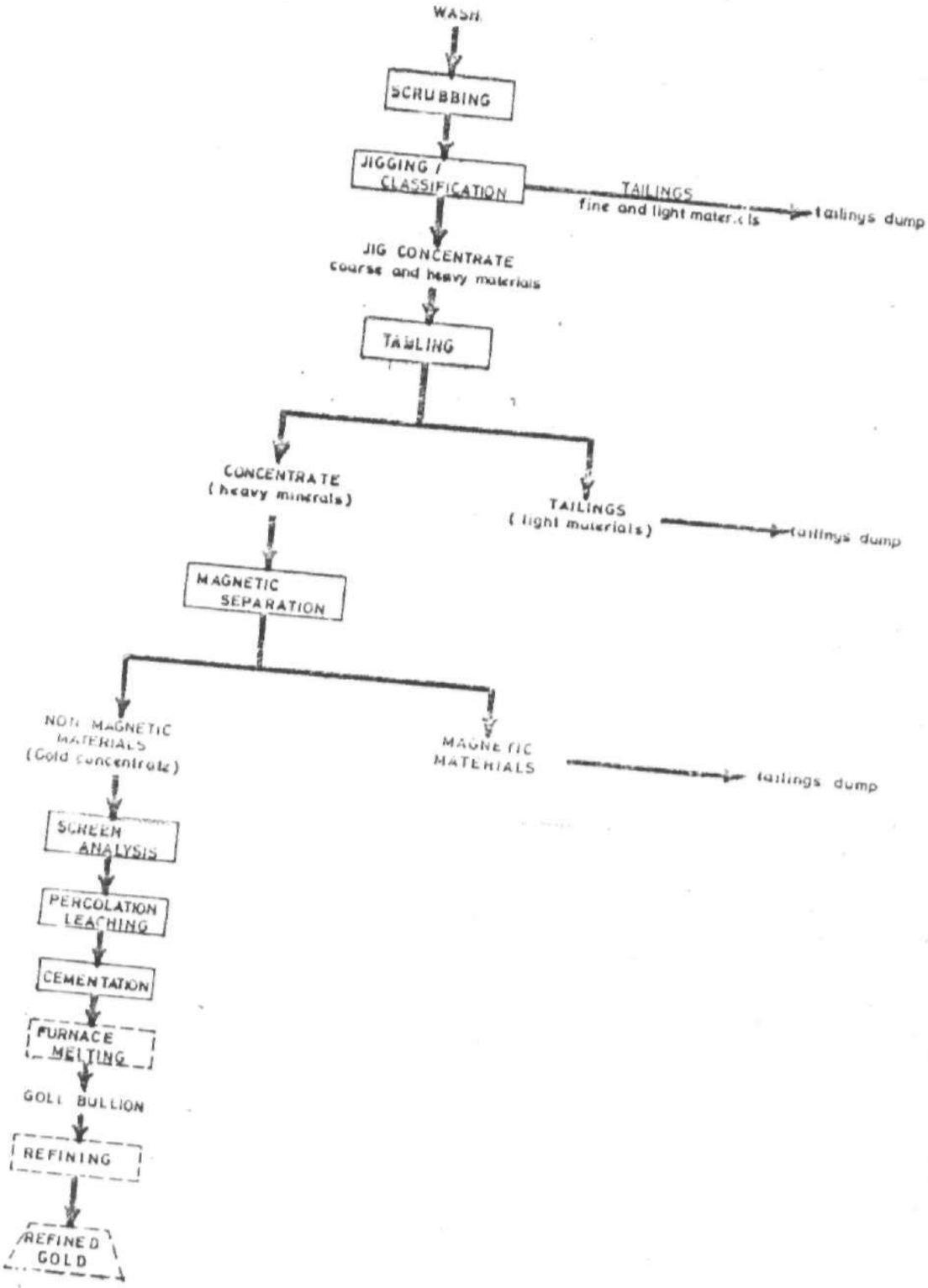


Fig. 4.12 Flowsheet development based on experimentation (percolation leaching).

cyanide solution obtained from cassava (Manihot esculanta), it was observed that yields of gold from cyanide solutions from cassava are relatively lower than those obtained from equivalent concentrations of standard NaCN. Even then, there is the possibility of augumenting if not replacing the analytical grade cyanide solutions with those obtained from cassava if the compounds in the solution inhibiting gold dissolution are removed or lowered in content. Thus a tropical enviroment like Nigeria which favours cassava production provides a ready source of cyanide for gold extraction and this concretizes the basis for the choice of cyanidation (by percolation leaching) for extraction of gold from Igun ore deposit in Ilesha goldfield.

4.5.2 Flowsheet Development Based On Heap Leaching

Low grade ore and small tonnages are best treated by low-cost method such as dump or heap leaching. Since Ilesha gold concentrate is amenable to percolation leaching, its wash should be amenable to heap leaching if the operating parameters are optimized. Thus the flowsheet based on heap leaching shown in Figure 4.13 in Appendix 11 is proposed.

4.5.3 Tailings Management / Enviromental Regulation

The enviromental degradation effects of cyanidation cannot be discountenanced and a waste management programme based on total containment concept, contigent on future treatability testwork should be adopted. This should indicate the best treatment method to ensure that pond discharges would be in compliance with Federal Environmental Protection Agency (FEPA) requirements.

Recently, about twelve processes for cyanide removal or regeneration are available and several of these processes offer satisfactory results for specific effluents (Bhappi, 1990). Remarkable among these processes is the Inco Process (Conrad, 1984). This process utilizes SO_2 - air mixture as oxidant,

lime for pH control and presence of copper in solution as a catalyst. The Inco Process was reported to have met all regulation requirements and is currently adopted in several cyanidation plants in Canada. This may be experimented on Ilesha gold ore vis-a-vis FEPA requirements.

4.5.4 Proposed Process Flowchart

By combining the flowsheet in Figure 4.1 (based on production of alluvial gold concentrate from the wash); flowsheet in Figure 4.12 based on percolation leaching and flowsheet in Figure 4.13 based on heap leaching - inferred approach; the proposed process flowchart for Ilesha alluvial gold is obtained as shown in Figure 4.14.

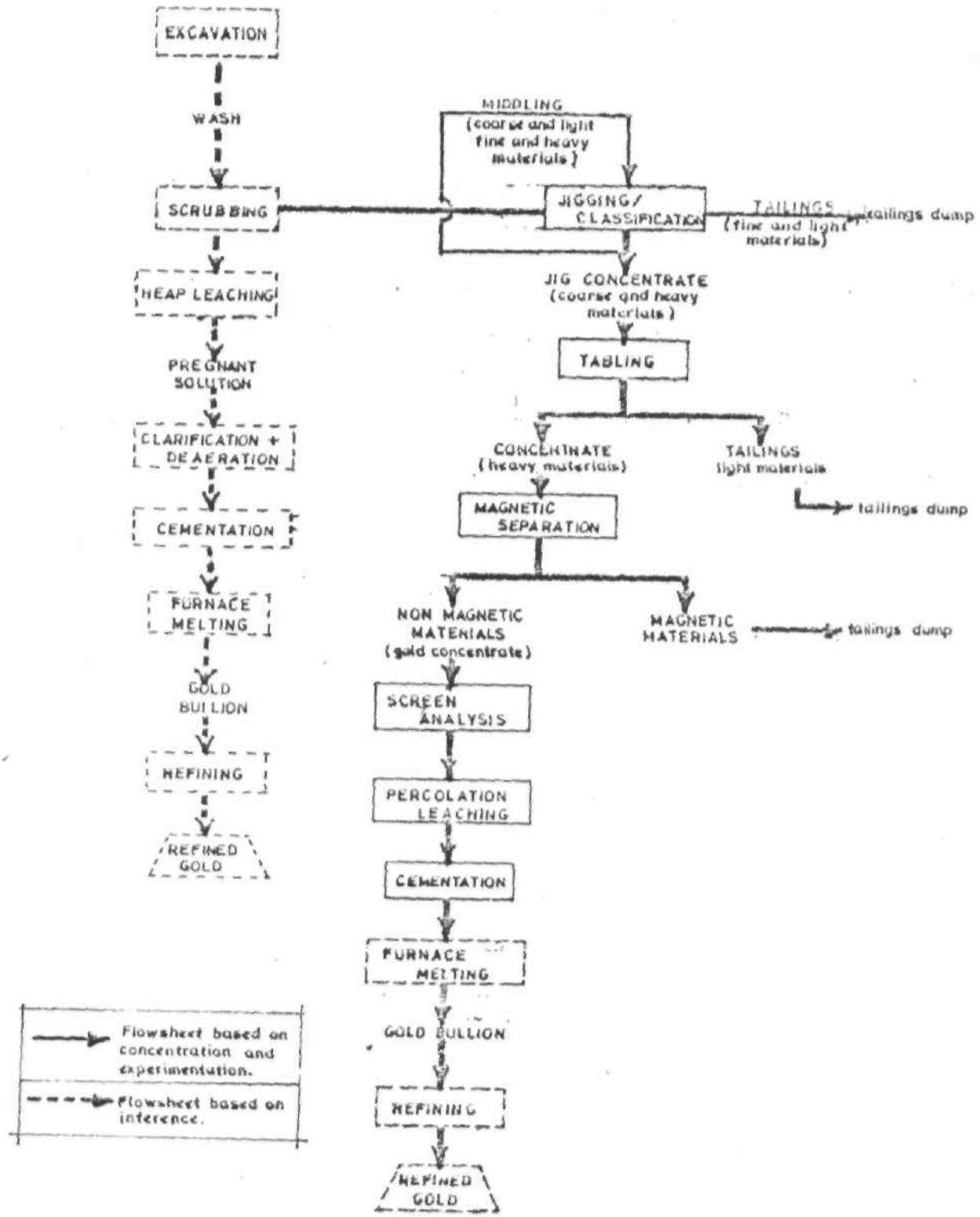


Fig.4.14 Proposed process flowchart for Igun Alluvial Gold Ore.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATIONS

The whole exercise of mineral processing, mineralogical characterization, ore chemical analysis and amenability tests on Ilesha alluvial gold ore has led to the evolution of the proposed process flowchart shown in Figure 4.17. When adopted, and if adapted, and carefully developed the process flowchart should engender substainable gold production in Ilesha goldfield.

5.1 Conclusion

(1) The data obtained on the particle size analysis show that most of the gold values are contained in the -600 +300μm to -75μm. That is, most of the gold values are contained in the fine particle size fractions.

(2) The x-ray diffractograms of different particle sizes show that free gold occurs in a very scanty amount in the following size fractions : -850 +600μm, -600+300μm, -300 +150μm and -75μm. Free gold could still occur in +2000μm, -2000 +850μm and 150 +75μm but this was not detected.

(3) The data on petrographic studies show a general trend that gold occurs as inclusion in sulphide phase and this phase tends to increase with decrease in particle size. The deposit is finely disseminated comprising very fine-grained largely sub-microscopic gold leading to the conclusion that Ilesha gold deposit belongs to non-refractory class.

(4) The gold content determined by fire-assay-gravimetric technique and atomic absorption spectrophotometer are essentially the same. For AAS, gold contents obtained for particle sizes +2000μm, -600 +300μm and -75μm particle size fractions are 4.666ppm 0.9818ppm and 89.11ppm respectively. Also for these size fractions the fire-assay values are

9.2g/ton, 20.0g/ton and 152.8g/ton respectively. Gold is present in all the size fraction; the general trend is that the gold content increases with decrease in particle size.

(5) Investigations on the recovery efficiency of amalgamation show that Igun gold ore responded poorly to amalgamation.

(6) Ilesha alluvial gold ore with Igun ore deposit as paradigm is amenable to percolation leaching.

At the optimum leaching parameters of 6-hour retention time, sodium cyanide concentration of 0.3% and hydrogen cyanide concentration (pH) of 11.5, the highest recovery value of 63.60% was obtained with the smallest particle size of -75μm. With availability of cyanide locally through extraction from cassava, percolation leaching is promising.

5.2 Recommendations

(1) In the course of this work, briquettes of the different concentrate size fractions were prepared with a view to examining them on ore microscope and scanning electron microscope but functional electron microscopes could not be sourced. For complete mineralogical investigation on, and characterization of Ilesha gold ore, functional electron microscope should be sourced and utilized. Specifically, it is necessary to establish the form of occurence of zinc in the ore.

(2) Further research on extractive metallurgy of Ilesha gold ore need to be carried out on:

(a) Amenability assessment of percolation leaching on Ilesha gold ore should incorporate more leaching parameters such as the effects of pulp density, heap agglomeration and oxygen concentration on gold recovery efficiencies.

(b) Cementation reaction until gold precipitate is produced from pregnant solution by zinc powder.

(3) Research into tailings management and design of suitable heap-leach pad for Igun gold deposit should be embarked upon to ensure solution conservation and environmental protection.

(4) Alluvial deposit has been considered in this work; process design for Ilesha Iode deposit at Iperindo should be considered in future work.

(5) Future work should also consider economic aspect of industrial scale production of gold. This will involve the use of pilot plant.

(6) Research should commence on recovery of gold from the tailings dump.

(7) Talcose rocks, from which talc ($3MgO.S_{14}\ O_{12}.H_2O$) could be extracted, abound within Ilesha goldfield. Besides, cassiterite (SnO_2) and columbite $(Fe,Mn)_2O_6$ have been won along with alluvial deposit (De Swardt, 1956). Furthermore, it has been established that ilmenite ($FeO.TiO_2$) is the major consituent of the opaque minerals of Ilesha gold concentrate. Ilmenite is a source of titanium oxide and the latter is used in paint manufacturing. Thus investment on Ilesha goldfield is justified not only for the purpose of gld extraction but also for the judicious exploitation of the aforementioned associated minerals.

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APPENDIX 1

BOX FILE FOR INTERPRETATION OF X-RAY DIFFRACTION DATA

Published by the

Joint Committee on powder diffraction standards,

1601 Park lane, Swarthmore, Pennsylvania, 19801, U.S.A.

3-125

d	5.16	2.30	2.04	5.16	$Ca_3Al_2(OH)_{12}$	$3CaO{\cdot}Al_2O_3{\cdot}6H_2O$
I/I$_1$	100	100	80	100	Calcium Aluminum Hydroxide	(Hydrogrossular)

Rad. $CuK\alpha_1$ λ 1.5405 Filter Dia.
Cut off I/I$_1$ I/I cor.
Ref. Flint, J. Res. Nat'l B. Stds., 26 27 (1941)

Sys. Cubic S.G. Isld (230)
a_0 12.56 b_0 c_0 A C
α β γ Z D_x
Ref. Ibid.

ω $n_\omega\beta$ $\epsilon\gamma$ Sign
2V D n_D Color
Ref.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
5.16	100	211			
4.47	60	220			
3.27	60	321			
3.13	60	400			
2.81	60	420			
2.56	20	422			
2.46	40	510			
2.30	100	521			
2.22	20	440			
2.04	80	611			
1.99	20	620			
1.81	20	444			
1.74	40	640			
1.71	40	721			
1.68	60	642			
1.60	20	732			
1.17	40	800			
1.41	40	840			

3-0709 MINOR CORRECTION

24-87 d 3-0709	2.96	2.76	1.97	3.46	$Ca_5\left[(as,P,Si)O_4\right]_3(OH,Cl,F)$
I/I$_1$ 3-0709	100	60	60	90	Calcium Silicate Phosphate Sulfate Hydroxide — Ellestadite

Rad. Fe λ Filter No.
Dia. 114.6mm Cut off Coll.
I/I$_1$ visual d corr. abs.?
Ref. D. McConnell, Am. Min. 22 981 (1937)

Sys. Hexagonal S.G. C^2_{6h} - $P6_3/m$
a_0 9.53 b_0 c_0 6.91 A C 0.725
α β γ Z
Ref. Ibid.

ω 1.633 $\epsilon\gamma$ 1.630 Sign -
2V D 3.068 n_D Color Pale rose
Ref. Ibid.

Sample from Crestmore, California, USA.
An apatite-type mineral with S and Si replacing P.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
3.46	90	002	1.35	5	331
3.13	20	130	1.31	5	124
2.96	100-	121	1.29	20	502
2.80	40	112	1.47	10	304
2.76	60	300	1.45	20	151
2.64	30	200			
2.57	5	301			
2.33	5	122			
2.30	30	130			
2.15	10	131			
2.07	5	113			
1.97	60	222			
1.91	20	132			
1.84	40	141			
1.83	70	231			
1.81	10	140			
1.78	30	402			
1.73	30	004			
1.68	10	232			
1.54	5	250			

3-219

d	4.35	1.48	3.36	4.35	$CuSiO_3{\cdot}2H_2O(?)$	$CuO{\cdot}SiO_2{\cdot}2H_2O(?)$
I/I$_1$	100	83	50	100	Copper Silicate Hydrate — Chrysocolla	

Rad. $CuK\alpha_1$ λ 1.54050 Filter Dia.
Cut off I/I$_1$ I/I cor.
Ref. Weingarang, Thesis, Lehigh U.,(1939)[unp]

Sys. S.G.
a_0 b_0 c_0 A C
α β γ Z D_x
Ref.

ω $n_\omega\beta$ $\epsilon\gamma$ Sign
2V D n_D Color
Ref.

This pattern was obtained from the mineral chrysocolla which is said to be impure copper silicate dihydrate. Chrysocolla is very nearly amorphous. Accordingly, the lines obtained on the diffraction pattern are not well defined. Analysis of this sample gave a mole ratio of CuO 0.48: SiO_2 1.00: H_2O 1.00.
Locality not given.

d Å	I/I$_1$	d Å	I/I$_1$	d Å	I/I$_1$
4.35	100				
3.36	50				
2.92	20				
2.61	20				
2.46	20				
1.63	10				
1.48	80				
1.32	10				

3-0772 MINOR CORRECTION

24-73 d 3-0772	2.75	2.46	1.66	2.73	CoS_2	CoS_2
I/I$_1$ 3-0772	100	60	55	100	Cobalt Sulfide	Cattierite

Rad. λ ? Filter
Dia. Cut off Coll.
I/I$_1$ Photocell d corr. abs.?
Ref. Kehl, P.F., Am. Min. 30, 448 (1945)

Sys. Cubic S.G.
a_0 5.523 b_0 c_0 A C
α β γ Z 4 D_x 4.80
Ref. Kehl, P.F., Am. Min., 30, 448 (1945)

ω $n_\omega\beta$ $\epsilon\gamma$ Sign
2V D n_D Color
Ref.

Sample from Shinkolobwe Mine, Katanga, Belgian Congo.
Analysis (Anon.): 60.60 Co, 1.53 Ni, 3.97 Fe, 33.90 S

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.75	100	200			
2.46	60	210			
2.25	48	211			
1.95	34	220			
1.66	55	311			
1.59	17	222			
1.53	17	230			
1.47	22	231			
1.37	14	331			
1.24	6	420			
1.21	5	421			
1.18	3	332			
1.13	17	242			
1.06	55	333			

3-0402 MINOR CORRECTION

1468 d 3-0402	3.39	4.41	3.22	8.02	$BaFeSi_4O_{10}$	$BaO{\cdot}FeO{\cdot}4SiO_2$
I/I$_1$ 3-0402	100	70	70	40	Barium Iron Silicate - Gillespite -	

Rad. MoKα λ 0.709 Filter Zr
Dia. 16 inches Cut off Coll.
I/I$_1$ d corr. abs.?
Ref. Pabst, Am. Min., 22, 372-390 (1943)[in private communication]

Sys. Tetragonal S.G. D^9_{4h} $P4_2/nmc$
a_0 7.485 b_0 c_0 16.030 A C 2.142
α β γ Z 4
Ref. Pabst, Am. Min., 22, 372-390 (1943)

ω $n_\omega\beta$ $\epsilon\gamma$ Sign
2V D 3.402 n_D Color
Ref.

Dx 3.407
Samples from Dry Delta, Alaska Range, Alaska and near Inglis, Manitoba County, California, USA.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
8.02	40	002			
5.30	10	110	1.84	10	323
4.41	70	112			108
3.51	30	104	1.87	40	119
3.39	100	200	1.78	90	400
3.22	70	114	1.72	10	314
3.09	90	213	1.65	10	330
2.75	40	204	1.64	30	422
2.65	50	006			
			1.58	20	
2.51	80	105	1.48	20	
		221	1.45	80	
		116	1.38	30	
2.39	90	302	1.29	90	
		310			
2.27	30	312	1.19	10	
2.21	10	224	1.18	30	
		314	1.08	10	
2.03	40	322	0.995	10	
		008			

3-0781 MINOR CORRECTION

2741 d 3-0781	2.74	1.72	2.54	3.73	$FeTiO_3$	$FeO{\cdot}TiO_2$
I/I$_1$ 3-0781	100	100	85	50	Iron Titanium Oxide	Ilmenite

Rad. CoKα λ 1.7902 Filter Fe
Dia. 9 in. Cut off Coll.
I/I$_1$ Visual d corr. abs.? Yes
Ref. United Steel Companies, Sheffield

Sys. Rhombohedral[R] S.G. C^2_{3i} - $R\bar{3}$
a_0 5.523 b_0 c_0 A C
α 54.51° β γ Z D_x 4.79
Ref. United Steel Companies, Sheffield

ω $n_\omega\beta$ $\epsilon\gamma$ Sign
2V D n_D Color
Ref. T.c. 315°C

Natural mineral
From hexagonal axes: a_0 = 5.079, c_0 = 14.135

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
3.73	50	100	1.07	10	3110
2.74	100	104	1.00	30	318
2.54	85	110	0.978	50	1014
2.23	10	113	.970	10	3013
1.86	85	204			314
			.960	10	410
1.72	100	116	.921	85	3110
1.63	50	108	.913	10	2014
1.50	65	214			
1.47	85	300			
1.34	10	1010			
1.27	60	220			
1.20	30	312			
1.18	60	2010			
1.15	10	314			
1.12	10	226			

| | | |

d	2.03	1.17	1.43	1.01
I/I$_1$	100	30	20	100

$(Fe)_2O_3$

IRON (α PHASE) (IRON, ALPHA)

Rad. CuKα λ 1.5405 Filter Ni Dia.
Cut off Cu Kα_2
I/I$_1$ COUNTER DIFFRACTOMETER d corr. abs.?
Ref. SWANSON ET AL., NBS CIRCULAR 539 VOL. II, #3 (1954)

Sys. CUBIC S.G. O_h^7 - $Fd3m$ (227)
a_o 2.8664 b_o c_o A C
α β γ Z 8
Ref. Ibid.

ε α nωβ 2.87 ηγ Sign
2V D 5.274 mp Color
Ref. Ibid.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.7060	100	110			
1.4332	30	200			
1.1702	30	211			
1.0134	10	220			
0.9064	12	310			
.8275	8	222			

d	4.34	3.12	2.10	4.84
I/I$_1$	100	65	35	100

$(NH_4)_2SiF_6$

AMMONIUM SILICON FLUORIDE (CRYPTOHALITE)

Rad. CuKα$_1$ λ 1.5405 Filter Ni Dia.
Cut off I/I$_1$ DIFFRACTOMETER
Ref. NBS CIRCULAR 539, VOL. 5, P 5 (1955)

Sys. CUBIC S.G. O_h^5 - $Fm3m$ (225)
a_o 8.295 b_o c_o A C
α β γ Z 4 Dx 2.0031
Ref. Ibid.

ε α nωβ 1.3695(Na) ηγ Sign
2V D 2.011 mp Color COLORLESS
Ref. DANA'S SYSTEM OF MINERALOGY 7TH ED. VOL. 2

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
4.344	100	111	1.0496	2	800
3.145	35	200	1.0079	*1	550
2.467	18	220	0.9859	2	822
2.321	8	311	.9548	4	751
2.022	45	222	.9194	*1	840
2.048	35	400	.9210	*1	911
[illegible]	4	331	.8949	2	664
1.877	4	420	.8561	*1	564
1.713	19	422	.8210	2	10.1.0
1.615	4	440	.8113	2	951
1.4198	8	531			
[illegible]					
[illegible]					
[illegible]					
1.1623	2	640			
1.1210	2	642			
1.0934	4	731			

6-0710 MINOR CORRECTION

d	1.63	2.71	2.42	3.13
I/I$_1$	100	85	65	35

FeS_2

IRON SULFIDE (PYRITE)

Rad. CuKα λ 1.5405 Filter Ni Dia.
Cut off Coll.
I/I$_1$ DIFFRACTOMETER d corr. abs.?
Ref. SWANSON ET AL., NBS CIRCULAR 539 VOL. II (1955)

Sys. CUBIC S.G. T_h^6 - $Pa3$ (205)
a_o 5.417 b_o c_o A C
α β γ Z 4
Ref. Ibid.

ε α nωβ Fy Sign
2V D±5.01 mp Color BLACK (IN POWDER); BRASS YELLOW (IN CRYSTALS)
Ref. Ibid.

SPECT. ANAL. OF SAMPLES <0.1 % Al, Ca, Mg, Si; <0.01 % Co, Cu, Mo, Ni, Pb; <0.001 % Cr, Ge, Mn; <0.0001 % Ag.
X-RAY PATTERN AT 26°C.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
3.128	35	111	0.8565	5	620
2.709	65	200	.8281	4	533
2.423	65	210	.8166	4	622
2.2118	50	211	.7981	6	631
1.9155	40	220			
1.6332	100	311			
1.5640	16	222			
1.4525	20	230			
1.4448	25	321			
1.2427	12	331			
1.2113	14	420			
1.1823	8	421			
1.1545	8	332			
1.1057	8	422			
1.0427	25	511			
1.0060	8	432			
0.9892	6	521			
.9577	12	440			
.9090	16	600			
.8758	8	611			

7-17 MAJOR CORRECTION

d	4.10	3.25	4.05	7.01
I/I$_1$	100	55	45	10

$KAl(SO_4)_2 \cdot 12H_2O$

POTASSIUM ALUMINUM SULFATE HYDRATE (POTASH ALUM)

Rad. CuKα$_1$ λ1.5405 Filter Ni Dia.
Cut off I/I$_1$ DIFFRACTOMETER
Ref. NBS CIRCULAR 539 VOLUME 6 (1956)

Sys. CUBIC S.G. T_h^6 - $Pa3$ (205)
a_o 12.157 b_o c_o A C
α β γ Z 4 Dx 1.754
Ref. Ibid.

ε α nωβ 1.456 ηγ Sign
2V D 1.737 mp Color COLORLESS
Ref. Ibid. DANA'S SYSTEM OF MINERALOGY, 7TH ED. VOL. 2

SPECT. ANALYSIS SHOWS <0.01 % Cr, Mg, Pb, Si; <0.001 % Ba, Ca, Cs, Fe, Li, Na. α ALUM STRUCTURE TYPE. PATTERN MADE AT 25°C.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
7.02	10	111	1.119	8	822
5.44	40	210	2.015	8	432
4.96	20	211	2.055	-	531
4.293	100	220	1.027	10	600
4.050	45	221	2.000	4	610
3.667	12	311	1.973	8	611
3.250	55	321	1.934	16	620
3.039	25	400	1.899	4	621
1.950	20	410	1.877	A	541
2.466	14	411	1.855	4	533
2.789	35	331	1.833	4	622
2.715	18	420	1.811	2	630
2.654	12	421	1.791	4	631
2.503	10	332	1.718	2	543
2.481	8	422	1.702	4	711
2.385	14	431	1.686	4	640
2.310	10	511	1.670	4	641
2.259	4	430	1.654	2	721
2.200	8	521	1.626	8	642
2.150	2	440	PLUS 19 LINES TO 1.2038		

7-7 MAJOR CORRECTION

d	3.74	1.91	1.58	3.97
I/I$_1$	100	25	25	25

NH_4Cl

AMMONIUM CHLORIDE (SAL AMMONIAC)

Rad. CuKα$_1$ λ 1.5405 Filter Ni Dia.
Cut off I/I$_1$ DIFFRACTOMETER
Ref. NBS CIRCULAR 539, VOLUME 2, 59 (1953)

Sys. CUBIC S.G. O_h^1 - $Pm3m$ (221)
a_o 3.8756 b_o c_o A C
α β γ Z 1 Dx 1.527
Ref. Ibid.

ε α nωβ 1.641 ηγ Sign
2V D 1.532 mp Color COLORLESS
Ref. Ibid., DANA'S SYSTEM OF MINERALOGY, 7TH ED., VOL. 2

SAMPLE IS OF ACS PURITY. PATTERN MADE AT 26°C.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
3.87	25	100	0.7911	4	622
2.740	100	110			
2.238	4	111			
1.939	8	200			
1.733	6	210			
1.582	25	211			
1.370	6	220			
1.292	6	300			
1.225	6	310			
1.1687	4	311			
1.1188	2	222			
1.0751	2	320			
1.0359	4	321			
0.9690	2	400			
.9400	4	410			
.9134	4	411			
.8890	2	331			
.8667	2	420			
.8457	4	421			
.8263	4	332			

7-22 MAJOR CORRECTION

d	4.33	4.08	3.27	7.09
I/I$_1$	100	80	75	55

$NH_4Al(SO_4)_2 \cdot 12H_2O$

AMMONIUM ALUMINUM SULFATE HYDRATE (TSCHERMIGITE)

Rad. CuKα$_1$ λ 1.5405 Filter Ni Dia.
Cut off I/I$_1$ DIFFRACTOMETER
Ref. NBS CIRCULAR 539 VOLUME 6 (1956)

Sys. CUBIC S.G. T_h^6 - $Pa3$ (205)
a_o 12.240 b_o c_o A C
α β γ Z 4 Dx 1.642
Ref. Ibid.

ε α nωβ 1.458 ηγ Sign
2V D 1.645 mp Color COLORLESS
Ref. Ibid. DANA'S SYSTEM OF MINERALOGY 7TH ED. VOL 2

SPECT. ANALYSIS OF SAMPLE SHOWS <0.01 % Fe, Mg, Si, Sn; <0.001 % Ca. α ALUM STRUCTURE TYPE. PATTERN MADE AT 25°C.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
7.09	55	111	2.237	12	521
6.13	12	200	2.130	8	502
5.48	55	210	2.096	4	433
4.998	35	211	2.068	8	531
4.327	100	220	2.029	10	600
4.079	90	221	2.012	10	610
3.691	35	311	1.985	10	611
3.195	4	320	1.935	16	620
3.272	75	321	1.910	2	621
3.060	30	400	1.888	4	541
2.967	20	410	1.868	8	533
2.882	11	411	1.846	6	622
2.810	35	332	1.815	<6	630
2.738	18	420	1.795	2	631
2.672	14	421	1.758	<1	444
2.608	12	332	1.749	2	632
2.499	10	422	1.731	10	543
2.402	8	431	1.714	4	711
2.358	12	511	1.698	2	640
2.314	8	432	PLUS 29 LINES TO 1.2008		

4-0777

4-0777 MAJOR CORRECTION

d 4-0777	2.41	1.70	2.78	2.778	CaO
I/I_1 4-0777	100	45	34	34	Calcium Oxide , (Lime)

Rad. $CuK\alpha_1$ λ 1.5405 Filter Ni
Dia. Cut off 50 Coll.
I/I₁ G. C. Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, NBS (1950)

Sys. Cubic S.G. O_h^5 - $Fm3m$
a_0 4.8105 b_0 c_0 A C
α β γ Z 4
Ref. Ibid.

εα nωβ 1.837 εγ Sign
2V D 3.345 mp (RA) Color
Ref. (1935)

REG ANALYSIS SHOWS ABOUT 0.21% H_2O, 0.1% Na, AND NO OTHER IMPURITY OVER 0.04%
At 29°C
TO REPLACE 1-1160, 2-1088, 3-1123

d Å	I/I₁	hkl	d Å	I/I₁	hkl
2.778	34	111			
2.405	100	200			
1.701	45	220			
1.451	10	311			
1.390	5	222			
1.203	4	400			
1.1036	4	331			
1.0755	9	420			
0.9819	9	422			
.9258	3	511			
.8506	6	440			
.8131	5	531			
.8018	6	600			

4-778

d	2.40	3.11	1.65	6.98	KMn_8O_{16}
I/I_1	100	80	80	40	Potassium Manganese Oxide Cryptomelane

Rad. $FeK\alpha_1$ λ 1.93597 Filter Dia.
Cut off I/I_1 I/I
Ref. Mathieson and Nadsley, Melbourne, Australia

Sys. Monoclinic S.G. *
a_0 9.79 b_0 2.88 c_0 9.94 A 3.39 C 3.45
α β 90°37' γ Z D_x 4.33
Ref. Mathieson and Nadsley, Am. Min., 35 99-101 (1950)

εα nωβ εγ Sign
2V D 4.35 mp Color
Ref. Ibid.

*C_s^1 - Ia or C_{2h}^1 - I2/a
From the Manganese Mine, Sitapur, Chindwara Dist., India.
A tetragonal form is known to exist.
(Bystrom, Acta Cryst., 3 146 (1950)).

d Å	I/I₁	hkl	d Å	I/I₁	hkl
6.98	40	101,10$\bar{1}$	1.745	10	
4.97	30	002	1.723	10	
4.84	50	200	1.684	10	
3.514	10	20$\bar{2}$	1.654	80	
3.454	20	202	1.617	70	
3.149	30	10$\bar{3}$	1.553	10	
3.110	80	103	1.535	20	
3.097	30	30$\bar{1}$			
3.065	20	301			
2.481	30	004			
2.425	10	400			
2.399	100	Indexed			
2.366	5	by JVS			
2.194	40				
2.170	30				
2.149	30				
2.030	20				
1.907	20				
1.843	40				
1.820	50				

4-0783

4-0783 MAJOR CORRECTION

d	2.36	2.04	1.23	2.36	Ag
I/I_1	100	40	26	100	Silver (Silver)

Rad. $CuK\alpha$ λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I₁ G-M Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, NBS (1950)

Sys. Cubic S.G. O_h^5 - $Fm3m$
a_0 4.0861 b_0 c_0 A C
α β γ Z 4
Ref. Ibid.

εα nωβ εγ Sign
2V D nφ Color
Ref. D_x 10.500 (1935)

PATTERN MADE AT 25°C
TO REPLACE 1-1164, 1-1167, 3-0921, 3-0931, 3-1316, 2-1088

d Å	I/I₁	hkl	d Å	I/I₁	hkl
2.359	100	111			
2.044	40	200			
1.445	25	220			
1.231	26	311			
1.1796	12	222			
1.0215	4	400			
0.9375	15	331			
.9127	12	420			
.8361	13	422			

9-421 MINOR CORRECTION

d	2.86	2.93	2.49	4.58	(Pt,Pd,Ni)S
I/I$_1$	100	30	20	10	PLATINUM PALLADIUM SULFIDE — BRAGGITE

Rad. [illegible] Filter [illegible] Dia. 5".1mm
Cut off I/I$_1$ VISUAL I/I cor.
Ref. BERRY AND THOMPSON, GEOL. SOC. AM. MEM., 14 71 (1962)

Sys. TETRAGONAL SG D_{4h}^{14} - $P4_2/mmc$ (137)
a_o 7.571 b_o c_o 7.71 A C1.031
α β γ Z 8 Dx 8.39
Ref. IBID., BANNISTER, MIN. MAG. 23 148 (1932)

Fo nωε Fγ Sign
2V D 10 Color
Ref. DANA'S SYSTEM OF MINERALOGY, 7TH EDITION

Pt 4.5 Pt, 2.6 Pd, 1.4 Ni
SAMPLE FROM RUSTENBURG, TRANSVAAL, S. AFRICA.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
4.59	10	011,210	1.391	30	
3.75	5	111	1.375	5	
3.61	5	020	1.307	5	
2.93	30	013	1.269	5	
2.86	100	021,120	1.197	20	
2.69	5	112	1.178	5	
2.64	30	121	1.139	20	
2.58	10	122,221	1.126	10	
2.01	10	031,130	1.079	10	
1.852	30	212	1.037	30	
1.807	10	023	0.980	5	
1.771	10	230	.961	10	
1.731	10	123,132			
1.712	30	231			
1.642	10	004			
1.624	30	[illegible]			
1.603	5	[illegible]			

9-423 MINOR CORRECTION

d	2.02	1.35	1.59	2.02	$CuFeS_2$
I/I$_1$	100	80	60	100	COPPER IRON SULFIDE — CHALCOPYRITE

Rad. FEKC λ 1.9373 Filter MnO Dia. 57.3mm
Cut off I/I$_1$ VISUAL
Ref. R.S. FERGUSON AND W. A. PEACOCK, GEOL. SOC. AM. MEM. 85 30 (1962)

Sys. TETRAGONAL SG D_{2d}^{12} - $I\bar{4}2m$ (122)
a_o 5.280 b_o c_o 10.409 A C1.9716
α β γ Z 4 Dx 4.283
Ref. SUNDIUS AND ELLIS, J. AM. CHEM. SOC. 33 2519 (1917)

Fo nωε Fγ Sign
2V D 4.2 mp Color BRASS-YELLOW
Ref. DANA'S SYSTEM OF MINERALOGY, 7TH ED.

SAMPLE FROM CUMBERLAND, ENGLAND.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
3.08	100	112			
2.69	1	020,004			
1.969	40	220			
1.854	30	024			
1.791	40	132			
1.573	20	116,023			
1.518	1	008			
1.323	10	040			
1.302	5	006			
1.214	10	332			
1.205	20	136,143			
1.099	60	244			
1.069	30	208			
1.018	20	152			
1.014	10	336			
1.005	5	1,1,-10			

9-424 MAJOR CORRECTION

d	3.06	1.87	1.60	11.7	(Cu,Fe,Ge)(S,As)
I/I$_1$	100	80	60	5	COPPER, IRON, GERMANIUM ARSENIC SULFIDE — RENIERITE

Rad. $Cu_{K\alpha}$ λ Filter Dia.
Cut off I/I$_1$
Ref. SCLAR AND GEIER, ECON. GEOL. 52 612-31 (1957) FROM MURDOCH, AM. MIN. 38 796 (1953)

Sys. PSEUDOCUBIC SG $P\bar{4}3m$ (218)
a_o 10.560 b_o c_o A C
α β γ Z 32 Dx
Ref. IBID.

Fo nωε Fγ Sign
2V D mp Color
Ref.

SAMPLE FROM PRINCE LEOPOLD MINE, KIPUSHI, BELGIAN CONGO.
PATTERN CLOSE TO GERMANITE AND COLUSITE; ONLY RENIERITE IS OPTICALLY ANISOTROPIC.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
11.9	5	?	2.13	5	006
10.259	5	001	2.075	5	015,134
8.467	5	?	2.03	5	115
7.50	10	011	1.87	80	044
6.05	5	111	1.858	5	?
5.30	5	002	1.816	5	035,334
4.80	5	012	1.722	5	115,335
4.31	10	112	1.595	60	224
3.75	5	022	1.56	5	136
3.55	5	003	1.529	10	444
3.34	10	013	1.497	5	017
3.06	100	222	1.472	5	117,155
2.96	5	023	1.444	5	127,2254
2.82	10	123	1.435	5	?
2.65	20	004	1.395	5	445,0174
2.56	5	014	1.325	10	008
2.50	5	033,114	1.317	–	?
2.386	5	024	1.314	20	266
2.255	5	233	1.194	5	068
2.163	5	224	FILM 9 LINES TO 0.995Å		

9-425 MINOR CORRECTION

d	2.86	1.67	1.83	1.47	$CoCu_2S_4$ $CuCo_2S_4$
I/I$_1$	100	80	60	20	COBALT COPPER SULFIDE — CARROLLITE

Rad. FEKC λ 1.9373 Filter Mn Dia. 57.3mm
Cut off I/I$_1$ VISUAL I I cor.
Ref. BERRY AND THOMPSON, GEOL. SOC. AM. MEM., 14 77 (1962)

Sys. CUBIC SG O_h^7 - $Fd3m$ (227)
a_o 9.48 b_o c_o A C
α β γ Z 8 Dx 4.82
Ref. IBID.

Fo nωε Fγ Sign
2V D 4.5-4.8 mp Color LIGHT-GRAY
Ref. DANA'S SYSTEM OF MINERALOGY, 7TH ED.

SAMPLE FROM M'KAMA, N. RHODESIA.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
3.37	20	111	1.696	10	139
3.28	60	002			
2.98	100	113			
2.37	50	004			
1.824	30	224			
1.705	10	115,333			
1.674	80	044			
1.600	5	135			
1.501	10	026			
1.448	20	335			
1.369	30	444			
1.237	30	117,155			
1.187	30	046			
1.136	10	[illegible]			
1.118	10	[illegible]			
1.098	30	[illegible]			
1.061	5	119,357			
1.032	5	466			

9-426 MINOR CORRECTION

d	2.75	3.40	3.39	1.37	$Pb_{19}As_{10}S_{40}$ $13PbS.As_2S_3$?
I/I$_1$	100	30	10	10	LEAD ARSENIC SULFIDE — RATHITE I

Rad. CuKα λ 1.5418 Filter Ni Dia. 57.3mm
Cut off I/I$_1$ VISUAL
Ref. L.G.BERRY, QUEEN'S UNIVERSITY, KINGSTON, CANADA

Sys. MONOCLINIC SG C_{2h}^5 - $P2_1/a$ (14)
a_o 25.1 b_o 7.91 c_o 8.66 A 3.173 C 1.094
α 89°00' γ Z 1 Dx 5.31
Ref. IBID.

Fo nωε Fγ Sign
2V D 5.37 mp Color LEAD-GRAY
Ref. DANA'S SYSTEM OF MINERALOGY, 7TH ED.

SAMPLE FROM BINNENTAL, SWITZERLAND (USNM 34101)

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
7.37	10	110	1.938	?	
4.41	10	210	1.917	10	
3.75	5	310	1.895	30	
4.37	40	410	1.831	10a	
4.19	40	510,002	1.781	30	
3.97	30	020	1.728	20	
3.73	30	002,220	1.665	10	
3.60	80	310,112,320	1.599	?	
3.39	10	212,412	1.564	30	
3.23	?	402,312	1.552	?	
3.09	40	602,320	1.443	?	
2.97	80	412,512	1.398	10	
2.87	70	810,220,022	1.351	?	
		222,620	1.327	10	
2.79	100	712,222,620	1.283	?	
2.62	30	022,322,220	1.264	?	
2.30	40a		0.984	?	
2.22	50				
2.12	10				
2.04	?				

9-427 MAJOR CORRECTION

d	2.57	1.54	2.87	4.04	$Fe_2Al_2(SiO_4)_3$ $3FeO.Al_2O_3.2SiO_2$
I/I$_1$	100	50	40	30	IRON ALUMINUM SILICATE — ALMANDITE

Rad. CuKα λ 1.5405 Filter Ni Dia. 114.6mm
Cut off I/I$_1$ VISUAL
Ref. L.G. BERRY, QUEEN'S UNIVERSITY, KINGSTON, CANADA

Sys. CUBIC SG O_h^{10} - $Ia3d$ (230)
a_o 11.53 b_o c_o A C
α β γ Z 8 Dx 4.313
Ref. IBID.

Fo nωε 1.815 Fγ Sign
2V D 4.29 mp Color RED
Ref. IBID.

SAMPLE FROM CAVENDISH TP., PETERBOROUGH CO., ONTARIO, CANADA.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
4.04	30	220	0.947	5	12.2.0
2.873	40	400	.935	10	12.2.2
2.569	100	420	.869	5	12.4.4
2.447	5	332	.860	10	12.6.0
2.348	20	422	.850	5	12.6.2
2.257	20	510	.835	5	888
2.102	30	521	.792	20	14.4.0
2.043	10	440	.785	20	14.4.2
1.986	30	611			
1.880	30	444			
1.599	40	640			
1.540	50	642			
1.461	20	800			
1.387	20	840			
1.257	30	842			
1.229	10	664			
1.167	5	941			
1.090	20	10.4.0			
1.051	10	10.4.2			
1.019	10	880			

4-0777 MAJOR CORRECTION

d	2.61	1.70	1.78	2.778
I/I$_1$	100	45	34	34

CaO

Calcium Oxide (Lime)

Rad. CuKα_1 λ 1.5405 Filter Ni
Dia. Cut off 50 Coll.
I/I$_1$ G. E. Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, 1955 (1953)

Sys. Cubic S.G. O_h^5 - Fm3m
a_o 4.8105 b_o c_o A C
α β γ Z 4
Ref. Ibid.

ε α n_ω β 1.837 γ Sign
2V D 2.345 mp (Na) Color
Ref. (1955)

NBS analysis shows about 0.21% MgO, 0.1% Ba, and no other impurity over 0.04%
At 29°C
To replace 1-1160, 3-1089, 3-1123

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.778	34	111			
2.405	100	200			
1.701	45	220			
1.451	10	311			
1.390	5	222			
1.203	4	400			
1.1036	4	331			
1.0755	9	420			
0.9813	9	422			
.9258	3	511			
.8504	4	440			
.8131	5	531			
.8016	6	600			

4-778

d	2.40	3.11	1.65	6.98
I/I$_1$	100	90	80	40

KMn_8O_{16}

Potassium Manganese Oxide Cryptomelane

Rad. FeKα_1 λ 1.93597 Filter Dia.
Cut off I/I$_1$ I/I
Ref. Mathieson and Wadsley, Melbourne, Australia

Sys. Monoclinic S.G. -
a_o 9.79 b_o 2.88 c_o 9.94 A 3.39 C 3.45
α β 90°27' γ Z Dx 4.51
Ref. Mathieson and Wadsley, Am. Min., 35 99-101 (1950)

ε α n_ω β γ Sign
2V D 4.55 mp Color
Ref. Ibid.

*Cl_2^- - Ia or Cl_{2h}^5 - I2/a
From the Manganese Mine, Sitapar, Chindwara Dist., India.
A tetragonal form is known to exist.
(Bystrom, Acta Cryst., 3 146 (1950)).

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
6.98	40	101,10$\bar{1}$	1.745	10	
4.97	30	002	1.723	10	
4.84	50	200	1.684	10	
3.514	10	20$\bar{2}$	1.654	55	
3.454	20	202	1.617	70	
3.149	30	10$\bar{3}$	1.553	10	
3.110	80	103	1.535	20	
3.097	30	30$\bar{1}$			
3.045	20	301			
2.481	30	004			
2.425	10	400			
2.399	100	Indexed by JVS			
2.366	5				
2.194	40				
2.170	30				
2.149	30				
2.010	20				
1.907	20				
1.843	40				
1.820	50				

4-0783 MAJOR CORRECTION

d	2.36	2.04	1.23	2.36
I/I$_1$	100	40	26	100

Ag

Silver (Silver)

Rad. CuKα λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I$_1$ G-M Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, 1955 (1950)

Sys. Cubic S.G. O_h^5 - Fm3m
a_o 4.0862 b_o c_o A C
α β γ Z 4
Ref. Ibid.

ε α n_ω β γ Sign
2V D mp Color
Ref. D_x10.5053 (1955)

Pattern made at 27°C
To replace 1-1164, 1-1167, 3-0881, 3-0931, 3-1316, 2-1098

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.359	100	111			
2.044	40	200			
1.445	25	220			
1.231	26	311			
1.1796	12	222			
1.0215	4	400			
0.9375	15	331			
.9137	12	420			
.8341	13	422			

4-0784 MINOR CORRECTION

d	2.36	2.04	1.22	2.36
I/I$_1$	100	52	36	100

Au

Gold (Gold)

Rad. CuKα λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I$_1$ G. E. Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, 1955 (1953)

Sys. Cubic S.G. O_h^5 - Fm3m
a_o 4.0788 b_o c_o A C
α β γ Z 4
Ref. Ibid.

ε α n_ω β γ Sign
2V D_x19.30mp Color
Ref.

Sample purified at NBS laboratory and is about 99.99+% Au.
At 26°C
To replace 1-1172, 1-1174, 2-1095

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.355	100	111			
2.039	52	200			
1.442	32	220			
1.230	36	311			
1.1774	32	222			
1.0196	8	400			
0.9358	23	331			
.9130	22	420			
.8325	23	422			

4-0793 MAJOR CORRECTION

d	2.32	1.64	1.34	2.68
I/I$_1$	100	60	1*	3

NaF

Sodium Fluoride (Villiaumite)

Rad. CuKα λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I$_1$ G. E. Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, 1955 (1953)

Sys. Cubic S.G. O_h^5 - Fm3m
a_o 4.64 b_o c_o A C
α β γ Z 4
Ref. Ibid.

ε α n_ω β (Na) γ Sign
2V D 2.802 mp Color
Ref. Ibid.

Spec. anal. Si-V, Mn-Ag others only as traces.
At 26°C
To replace 1-1181, 1-1184, 2-1115

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.680	3	111			
2.319	100	200			
1.639	90	220			
1.399	2	311			
1.338	17	222			
1.1588	7	400			
1.0433	<1	331			
1.0363	12	420			
0.9458	8	422			
.8930	1	511			
.8193	3	440			

4-797

d	2.30	2.24	1.60	3.87
I/I$_1$	100	100	80	20

Pb_2Pd_5

Lead Palladium (Plumbopalladinite)

Rad. CuKα λ 1.1418 Filter ? Dia. ?
Cut off I/I$_1$ Visual I/I$_{corr.}$ = Yes
Ref. Nowotny, Schubert and Dettinger, Metallforschung, Bd. 1, 1/2 117-165 (1946)

Sys. Hexagonal S.G. C_{3v}^1 - $P6_3$ mc
a_o 4.4650 b_o c_o 5.7090 A C 1.2785
α β γ Z 1 Dx
Ref. Ibid., Wyckoff, "The Structure of Crystals," 1st ed.

ε α n_ω β γ Sign
2V D 12.4 mp Color
Ref. Ibid.

NiAs type.
Annealed at 800°C.
$Pd_{3.1}Pb_2$ (55.8 wt. % Pb)

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
3.87	20	1010	0.936	70	3034,4042
3.21	70	1011	.936	30	3141
2.86	30	0002	.926	50	1016
2.30	100	1012	.917	50	4042
2.24	100	1120	.901	30	2135
1.85	30	2021	.881	70b	1126,2244
1.76	60	1122	.859	30	3144
1.71	30	1013	.847	70b	1251,4150
1.60	80	2022	.808	50b	4151,1261
1.42	60	0004,2131	.799	80	1017,2134
1.36	30	2023			
1.30	80	2132			
1.29	60	3030			
1.21	80	1124			
1.18	30	3031			
1.15	30	2024,2133			
1.12	60	1240			
1.06	20	3141			
1.04	20	2242			
1.01	60	3142			

13-520

d	3.03	2.5	2.46	4.86
I/I$_1$	100	70	70	50

$(K,Na,Ca)_4(Fe^{2+},Fe^{3+},Mg,Mn)_2(Si_4O_{10})_2(OH,F)$ ○

POTASSIUM SODIUM IRON SILICATE HYDROXIDE — FERRARISITE

Rad. FeKα λ POSSIBLY +K Filter +Fe Dia. 5".0mm
Cut off I/I$_1$ VISUAL
Ref. M. D. TOMASHEV ET AL., TRUDY MINERALOG. MUZEYA AKAD NAUK, SSSR no. 9 152-157 (1959). Am. Min. 45 752 (1960)

Sys. MONOCLINIC S.G. C2/m (12)?
a 14.95 b 4.98 c 9.79 A 2.142 C 1.403
α — β 112° γ Z 2 Dx
Ref. Ibid.

Na 1.541 (Na) n$_w$β 1.560 nγ 1.587 Sign +
2V 84° D 2.76 mp Color LIGHT ROSE
Ref. Ibid. (2 VOICES INDICATE 2V = 61°)

SAMPLE FROM A PEGMATITE IN THE KHIBINA TUNDRA, USSR.
2 ANALYSES GIVEN.
POOR AGREEMENT BETWEEN MEASURED AND CALCULATED D VALUES (1.55)

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
4.86	50	200,201	1.961	10	INDEXED BY LCR
5.21	20	111	1.875	60	
6.47	50	202	1.835		
4.68		002	1.752	40	
6.10	40	112	1.690	20	
3.55	90	312	1.574	30	
3.44		402	1.559	40	
3.35	50	113	1.484	40	
3.25	50	021,203	1.447	50	
3.03	100	003	1.375	50	
2.88	60	403	1.360	10	
2.71	60	511	1.3---	10	
2.62	50	111	1.300	20	
2.52	20	421	1.258	20	
2.46	70	420,203	1.177	10	
2.29	10	130,023	1.119	30	
2.19	30	131	1.085	30	
2.10	10	111			
2.05	10	204			
1.995	20				

13-529

d	3.20	2.70	4.13	6.13
I/I$_1$	100	100	80	80

$Bi_4(\ \ )_3$ *i*

BISMUTH SILICATE (EULYTITE)

Rad. λ Filter Dia.
Cut off I/I$_1$
Ref. DUMIT-VARAMBON, BULL. SOC. FRANC.MIN. CRIST. 83 285 (1959)

Sys. CUBIC S.G. I$\bar{4}$3d (220)
a 10.30 b c A C
α β γ Z 4 Dx 6.76
Ref. IBID.; MENZER, Z. KRIST. 78 147 (1931)

Na n$_w$β nγ Sign
2V D mp Color
Ref.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
4.13	80	211	1.230	50	653
3.20	100	310			
2.70	100	312			
2.520	30	400			
2.070	70	422			
1.990	70	501			
1.876	40	521			
1.759	60	530			
1.664	70	611			
1.624	50	620			
1.584	70	541			
1.516	60	631			
1.483	30	444 ---			
1.452	90	550,702			
1.424	20	640			
1.399	60	554,721			
1.374	50	642			
1.349	40	730			
1.306	50	732			
1.266	70	811			

13-530 MINOR CORRECTION

d	4.79	4.02	5.05	7.9
I/I$_1$	100	90	50	5

$Fe^{2+}\ Fe_2^{3+}(SO_4)_4 \cdot 14H_2O$ *i*

IRON SULFATE HYDRATE — ROZENITE, ROMERITE

Rad. FeKα λ 1.9373 Filter Mn Dia. 57.3mm
Cut off I/I$_1$ VISUAL
Ref. VAN LOAN AND NUFFIELD, CAN. MINERAL. 6 368 (1959)

Sys. TRICLINIC S.G. P$\bar{1}$ (2)
a 6.68 b 15.26 c 6.27 A 0.425 C 0.411
α 90°04' β 100°45' γ 85°28' Z 1 Dx 2.189
Ref. Ibid.

Na 1.520 n$_w$β 1.570 nγ 1.578 Sign –
2V D 2.18 mp Color GLOVE BROWN
Ref. Ibid.

SAMPLE FROM ISLAND MTN., TRINITY COUNTY, CALIFORNIA, USA.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
7.9	5	020	1.740	5	INDEXED BY LCR
6.33	5	100			
5.05	50	030			
4.79	100	130,001			
4.22	10	101			
4.03	90	130,111			
3.66	10	121			
3.26	5	041			
3.16	20	200			
3.04	10	050			
2.94	5	201			
2.84	10	112,230			
2.62	20	132,032			
2.55	10	151,132			
2.38	30	160,042			
2.21	5	142,222			
2.11	5	201,310			
1.990	10	113,113			
1.907	20	162,033			
1.812	10				

13-533 MINOR CORRECTION

d	2.62	1.55	2.73	6.58
I/I$_1$	100	80	70	35

$CaAl_2Si_2O_7(OH)_2 \cdot H_2O$ *i*

CALCIUM ALUMINUM SILICATE HYDROXIDE HYDRATE — LAWSONITE

Rad. CuKα λ Filter Ni Dia. 114.6mm
Cut off I/I$_1$
Ref. DAVIS AND PABST, AM. J. SCI. 258 689 (1960)

Sys. ORTHORHOMBIC S.G. Bmm (62)
a 8.788 b 13.129 c 5.844 A 0.6694 C 0.4453
α β γ Z 4 Dx 3.094
Ref. IBID. PABST, Z. KRIST. 115 307 (1961)

Na 1.665 n$_w$β 1.673 nγ 1.686 Sign +
2V 79-85° D 3.09 mp Color
Ref. Ibid.

SAMPLE FROM TIBURON PENINSULA, MARIN CO., BLAKE GARDENS, CONTRA COSTA CO., CALIFORNIA, USA.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
6.58	35	020	2.053	5	341
4.84	60	101	1.990	5	141
4.57	30	111	1.965	30	430
4.18	40	210	1.894	15	113
3.91	5	121	1.852	5	351
3.64	60	220	1.786	5	251
3.28	35	040,131	1.752	35	042
2.923	45	002	1.728	20	370
2.728	90	141	1.697	15	422
2.680	50	022	1.685	5	450,303
2.624	100	240,101	1.657	5	143
2.573	5	231	1.631	20	521,432
2.433	40	202,321	1.626	40	242
2.395	30	212	1.575	10	323,531
2.311	15	151	1.550	80	440
2.284	20	222	1.501	10	541
2.254	35	150	1.444	40	004
2.190	15	400,060			
2.129	60	232			
2.089	5	420			

13-534 MAJOR CORRECTION

d	2.69	1.69	2.51	3.66
I/I$_1$	100	60	50	25

α-Fe_2O_3 ★

IRON OXIDE (HEMATITE)

Rad. CoKα λ 1.7902 Filter – Dia. 114.6mm
Cut off I/I$_1$ WOLL RECORDING MICROPHOTOMETER
Ref. RAVINDRANATHAN AND ALI, COUNCIL OF SCI. AND INDUST. RES., CENTRAL FUEL RESEARCH INSTITUTE, BIHAT, INDIA.

Sys. RHOMBOHEDRAL S.G. R3c (167)
a 5.0340 b c 13.752 A C
α β γ Z 6 Dx 5.271
Ref. KASTALSKY ET AL., AUST. J. CHEM., 21 1081 (1968)

Na 2.94 n$_w$β 3.22 nγ Sign –
2V D 5.26 mp 1350-1360 Color Dark-brown
Ref. IBID.; DANA'S SYSTEM OF MINERALOGY, 7TH ED.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
3.66	25	012	1.102	16	226
2.69	100	104	1.076	2	042
2.51	50	110	1.055	18	2110
2.285	2	006	1.042	2	1112
2.201	30	113	1.038	2	404
2.070	3	202	0.9890	10	232,318
1.838	40	024	.9715	2	229
1.690	60	116	.9601	18	324
1.634	5	211	.9578	5	0114
1.596	16	018	.9518	12	140,0213
1.481	35	214	.9354	5	413
1.452	35	300	.9204	4	048
1.349	4	208	.9080	25	1310
1.310	20	1010,119			
1.258	8	220			
1.228	2	036			
1.213	4	223			
1.189	8	128			
1.160	10	0210			
1.141	12	134			

13-535

d	3.71	3.35	3.22	11.6
I/I$_1$	100	90	80	50

$Ca_4BeAl_2Si_4O_{14}(OH)_2$ *i*

CALCIUM BERYLLIUM ALUMINUM SILICATE HYDROXIDE — DANBURITE

Rad. CuKα λ 1.5418 Filter Ni Dia. 114.6mm
Cut off 50 I/I$_1$ VISUAL
Ref. SWITZER AND REICHEN, AM. MINERAL. 43 757 (1960)

Sys. ORTHORHOMBIC S.G.
a 9.71 b 11.82 c 4.90 A 0.836 C 0.422
α β γ Z 1 Dx 2.81(calc) 2.97(obs)
Ref. USBM CLEVENGER, MIN. MAG. 23 495 (1940) GIVES (001) 10.19, (201) 9.11, (210) 8.76

Na 1.586 n$_w$β nγ 1.590 Sign
2V D 2.73 mp Color
Ref. Ibid.

SAMPLE FROM STRZEGOM, SILESIA (USNM R-7553 ORIGINALLY MINED MILKWHITE), POLAND.
ANALYSIS %: 5.52 BeO, 0.40 Fe_2O_3, 9.62 Al_2O_3, 23.44 CaO, 47.04 SiO_2, 2.86 H_2O^+, 0.14 H_2O^-, 0.22 MnO.
NEAR n=0.1, mn1.46. OTHER ANALYSES INDICATE THE GENERAL FORMULA WITH n=0.06 TO 0.86 mn1.74-1.46

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
11.1	50	010	2.36	70	231
9.83	10	100	2.48	50	041,240
7.44	50	110	2.42	50	141,002
6.98	50	120	2.37	30	410,012
6.65	60	200	2.32	60	050
4.82	10	111	2.29	40	112
4.35	10	101	2.26	40	150
4.21	40	111*	2.23	30	420,022
3.85	30	030	2.16	10	241
3.71	100	021,220	2.09	50	051,250
3.58	10	130	2.08	50	132*
3.40	10	201*	1.97	80	341
3.35	90	211*	1.95	60	500,302
3.22	80	300	1.87	40	042,440
3.12	80	310	1.82	60	501*
3.09	10	031,230			
2.85	60	221			
2.79	20	140			
2.73	20	301*			
2.65	10	311			

4-0777 MAJOR CORRECTION

d 4-0777	2.41	1.70	1.38	1.208
I/I$_1$ 4-0777	100	45	34	34

CaO

Calcium Oxide (Lime)

Rad. CuKα_1 λ 1.5405 Filter Ni
Dia. Cut off 50 Coll.
I/I$_1$ G. C. Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, 1955 (1950)

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.778	34	111			
2.405	100	200			
1.701	45	220			
1.451	10	311			
1.390	5	222			
1.203	4	400			
1.1036	4	331			
1.0795	9	420			
0.9819	9	422			
.9258	3	511			
.8504	4	440			
.8131	5	531			
.8018	6	600			

Sys. Cubic S.G. O_h^5 - $Fm3m$
a_o 4.8105 b_o c_o A C
α β γ Z 4
Ref. Ibid.

ε α n ω β 1.837 γ Sign
2V D 3.345 mp (N$_a$) Color
Ref.

NBS analysis shows about 0.21% MgO, 0.17% SiO$_2$, and no other impurity over 0.04%.
At 29°C
To replace 1-1160, 2-1089, 3-1153

d	2.40	3.11	1.65	6.98
I/I$_1$	100	80	80	40

KMn_8O_{16}

Potassium Manganese Oxide Cryptomelane

Rad. FeKα_1 λ 1.93597 Filter Dia.
Cut off I/I$_1$ I-I
Ref. Mathieson and Hadsley, Melbourne, Australia

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
6.98	40	101,101	1.745	10	
4.97	30	002	1.723	10	
4.84	50	200	1.684	10	
3.514	10	202	1.654	50	
3.454	20	202	1.617	70	
3.149	30	10$\bar{3}$	1.553	10	
3.110	80	103	1.515	20	
3.097	30	10$\bar{3}$			
3.061	20	301			
2.481	30	004			
2.425	10	400			
2.399	100	Indexed by JVS			
2.366	5				
2.194	40				
2.178	30				
2.149	30				
2.030	20				
1.907	20				
1.843	40				
1.820	30				

α n ω β γ Sign
2V D 4.35 mp Color
Ref. Ibid.

*C$_h^4$ - I4$_1$/a or C$_{4h}^6$ - I2/a
From the Manganese Mine, Sitapar, Chindwara Dist., India.
A tetragonal form is known to exist.
(Bystrom, Acta Cryst., 3 146 (1950)).

4-0784 MINOR CORRECTION

d 4-0784	2.36	2.04	1.27	2.36
I/I$_1$ 4-0784	100	52	36	100

Au

Gold (Gold)

Rad. CuKα λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I$_1$ G. C. Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, 1955 (1953)

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.355	100	111			
2.039	52	200			
1.442	32	220			
1.230	36	311			
1.1774	12	222			
1.0196	6	400			
0.9358	23	331			
.9120	22	420			
.8325	23	422			

Sys. Cubic S.G. O_h^5 - $Fm3m$
a_o 4.0785 b_o c_o A C
α β γ Z 4
Ref. Ibid.

ε α n ω β γ Sign
2V D_x19.3 mp Color
Ref.

Sample purified at NBS laboratory and is about 99.99% Au.
At 26°C
To replace 1-1172, 1-1174, 2-1095

4-0793 MAJOR CORRECTION

d 4-0794	2.32	1.64	1.34	2.68
I/I$_1$ 4-0793	100	40	1*	3

NaF

Sodium Fluoride (Villiaumite)

Rad. CuKα λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I$_1$ G. C. Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, 1955 (1953)

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.680	3	111			
2.319	100	200			
1.639	60	220			
1.399	3	311			
1.338	17	222			
1.1588	7	400			
1.0433	41	331			
1.0363	12	420			
0.9458	8	422			
.9320	1	511			
.8195	3	440			

Sys. Cubic S.G. O_h^5 - $Fm3m$
a_o 4.64 b_o c_o A C
α β γ Z 4
Ref. Ibid.

ε α n ω β (Na) γ Sign
2V D_x2.802 mp Color
Ref. Ibid.

Spec. anal. Si-V, mean others only as traces.
At 26°C
To replace 1-1181, 1-1194, 2-1115

4-0783 MAJOR CORRECTION

d	2.36	2.04	1.23	2.36
I/I$_1$	100	40	26	100

Ag

Silver (Silver)

Rad. CuKα λ 1.5405 Filter Ni
Dia. Cut off Coll.
I/I$_1$ G-M Diffractometer d corr. abs.?
Ref. Swanson and Tatge, JC Fel. Reports, 1955 (1950)

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
2.359	100	111			
2.044	40	200			
1.445	25	220			
1.231	26	311			
1.1796	12	222			
1.0215	4	400			
0.9375	15	331			
.9137	12	420			
.8341	13	422			

Sys. Cubic S.G. O_h^5 - $Fm3m$
a_o 4.0862 b_o c_o A C
α β γ Z 4
Ref. Ibid.

ε α n ω β γ Sign
2V D mp Color
Ref. 9×10^{-5}003 (1955)

Pattern made at 27°C
To replace 1-1184, 1-1167, 3-0921, 3-0931, 3-1316, 2-1098

4-797

d	2.30	2.24	1.60	3.87
I/I$_1$	100	100	60	30

Pb_2Pd_3

Lead Palladium (Plumbopalladinite)

Rad. CuKα λ 1.5418 Filter ? Dia. ?
Cut off I/I$_1$ Visual I/I$_{cor.}$ = Yes
Ref. Nowotny, Schubert and Dettinger, Metallforschung, Bd. 1, 4/5 137-145 (1946)

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
3.87	30	1010	0.936	70	1014,4041
3.21	70	1011	.936	50	3143
2.86	30	0002	.926	50	1016
2.30	100	1013	.917	50	4042
2.24	100	1120	.901	30	2135
1.83	30	2021	.881	70b	1126,2144
1.76	60	1122	.859	30	3144
1.71	30	1013	.847	70b	3252,4
1.60	60	2022	.808	50b	4152,1
1.42	60	0004,2131	.799	80	1017,2136
1.36	30	2023			
1.30	80	2132			
1.29	60	3030			
1.21	80	1134			
1.18	30	3032			
1.15	30	2024,2133			
1.12	60	2240			
1.06	20	3141			
1.04	20	2242			
1.01	60	3142			

Sys. Hexagonal S.G. C_{3v}^5 - $P6_3mc$
a_o 4.4650 b_o c_o 5.7090 A C 1.2781
α β γ Z 1 Dx
Ref. Ibid., Wyckoff, "The Structure of Crystals," 1st ed.

ε α n ω β γ Sign
2V D 12.4 mp Color
Ref. Ibid.

NiAs type.
Annealed at 800°C.
$Pd_{3.1}Pb_2$ (11.8 wt. % Pb)

APPENDIX 2

GOLD'S GENERAL PROPERTIES, ITS VARIOUS TESTS AND CHEMICAL FORMULAE OF ITS ASSOCIATED MINERALS AND GANGUES

Ores of gold include calaverite ($AuTe_2$) and silvernite (Au, Ag) Te_2.

Chemical Symbol, Au (from latin "Aurum") meaning shining down.

Atomic weight - 197.967

Atomic Number - 79

Melting point - 1,064.43^{0}C

Boiling point - 2660^{0}C

Specific gravity - 14 to 19.3 depending on purity

Valency - 1 or 3

No. of isotopes - 18

Colour - yellow when pure

Lustre - metallic

Streak - Yellow with metallic shine

Hardness - 2.5 to 3, malleable and can easily cut

Cleavage - None

Fracture - Hooked

Crystal System - Cubic

Habit - Crystals are rare, commonly spongy or leafy. Massive grains and scales are also usual

Reactivity - Noble, inert and unattacked by acids except aqua regia

Oxides - Au_2O_3, Au_2O

Geochemistry - Gold occurs with silver and copper in group 1B of the periodic table accounting for their common association in mineral deposits. The three elements have similar electronic structure with 's' electrons outside a filled 'd' electron orbital. However, gold has a siderophile nature whereas

copper and silver are chalcophile elements.

Distinguishing Features - Colour, specific gravity and softness. Insoluble in nitric acid and can be distinguished from pyrite (FeS) - 'fool's gold' - which is brittle, and chalcopyrite (CuFeS) which crumbles when cut with a knife. Methods of gold analysis include field tests, fire-assay and instrumental analysis.

Field Test for Gold

The most common field tests for gold include the following:

(i) Acid Test

(ii) Specific Gravity Test

(iii) Colour and Streak Test.

Acid Test: Acid Test is the simplest and quickest method of testing for gold¿ It involves the use of analytical grade nitric acid (HNO_3) which has the ability to dissolve all metals except gold and platinum. If on addition of gold to the acid solution, the colour of the acid changes, then the sample is neither pure gold nor gold at all.

Generally, if the sample is yellow, metallic, heavy and does not dissolve in nitric acid, then it is gold.

Specific Gravity Test: Colour and streak to an experienced gold dealer may be enough for identification of pure gold.

Pure gold is golden yellow and gives a shinning golden yellow streak. There are three other main colours of gold resulting from dilution of gold by alloying elements such as silver, copper, bismuth, zinc, nickel and palladium. For example gold may be pale yellow due to silver, rose-red due to copper and bismuth, white due to palladium and green due to nickel.

For such alloyed gold identification requires a combination of tests for various properties. The colour test involves a simple compesation of the colour and streak of the sample with the standard colours and streaks.

Fire-Assay Test: This is the most expensive but fool-proof test for gold.

This best method is particularly useful in determining the gold content of 'fool's gold' (that is, pyrite) and other minerals suspected to contain gold.

The fire-assay of gold is based mainly on the following principles:

1. Gold has a high solubility in molten lead but are almost completely insoluble in slags of suitable composition.

2. There is an appreciable difference in the specific gravity of the precious metal bearing alloys and slags in the liquid state which allows for complete and clear separation of the two products.

3. Lead is practically completely separated from the precious metal by carefully controlled oxidising fusion and subsequent absorption in a porous refactory vessel known as cupel.

4. Gold is completely insoluble in nitric acid.

The test entails fusion of the sample with borax, sodalime and silica flux using lead or nickel as gold collector, this is followed by cupellation. The fire-assay may be combined with gravimetric analysis or atomic absorption spectrophotometric (AAS) analysis in order to determine the gold content of the sample.

Instrumental Analysis: Atomic Absorption Spectrophotometer

Atomic absorption spectrophotometer (AAS) offers a useful technique for the determination of small quantities of gold. The selective atomic absorption following a concentration step of solvent extraction or fire-assy, enables a very low content of gold to be measured. The solvent extraction of gold bromine or chloride into methyl isobutyl ketone, and the aspiration of the latter into the flame, is one of the techniques.

Schroedinger explains the quatum theory which basically states that minute particles can only gain or lose energy in small finite units called quanta (packets of energy). It is used to obtain line spectra from excited

atoms. If an electron loses energy it will move towards the nucleus. Since this energy changes are quantized, these movements will be in jumps and they involve the absorption or emission of a quantum of energy characteristic of the change or transition. Energy is absorbed or emmited as light. The energy related to the wavelength of that light spectrum will be obtained.

In the AAS the line spectrum from this energy transition is used to determine the concentration of an element in solution. To do this, we produce in the flame a population of atoms in the ground state, through which we pass light of such a wavelength that the atoms will absorb it and out to an excited state. The amount of light absorbed will be a measure of the concentration of atoms in the flame and therefore concentration of atoms in the solution. The amount of radiation absorbed by a sample is described by Beer-Bourguer-Lambert law:

$$A = \varepsilon.b.c$$

where

A = absorbance in gl^{-1} (read on scale)

ε = molar absorptivity of the sample

b = path length (distance through which radiant energy has passed in the solution)

c = concentration of species

Chemical Formulae of Minerals Associated with Igun Ore Deposit and Its Gangues

Minerals	Chemical Formulae
Ilmenite	$FeO.TiO_2$
Geothite	FeOOH
Feldspar (Plagioclase)	$(Na,Ca)_4[(Al,Si)_4O_8](Cl,Co_3)$
Quartz	SiO_2
Hematite	α-Fe_2O_3
Pyrite	FeS_2
Renierite	(Cu,Fe,Ge)(S,As)
Barium Sulphate	$BaSO_4$
Chrysocola	$CuO.SiO_2.2H_2O$
Rathite	$Pb_{13}As_{12}S_{40}$
Ellestadite	$Ca_5(Si, P, SiO_4)_3(OH,Cl,F)$
Chalcopyrite	$CuFeS_2$
Gypsum	$CaCO_3.2H_2O$
Almadine	$Fe_3Al_2(SiO_4)_3$
Catterite	CoS_2
Lime	CaO

APPENDIX 3

MEASUREMENT OF GOLD PURITY, CONVERSION FACTORS AND STRATEGIC IMPORTANCE OF GOLD

CARAT: The most common measure of the purity of gold is known as carat (ct) (Karat in U.S.A.). It is used to state the proportion of gold in an alloy. Pure gold is 24 carat; that is 100% Au. In England, pure gold (24 ct) and from gold-alloys of 22, 18, 14 and 9 carat are legal 'tender. In the U.S.A. four gold alloys of 22, 18, 14 and 10 karats are legal tender.

FINENESS: Another measure of purity of gold is fineness; that is, parts of Au per 1000. The proportion of pure gold or silver jewellery, bullion or coin, often expressed in parts per thousand. It is commonly used to describe the purity of raw gold such as nuggets of native gold.

CONVERSION FACTORS

From	To	Multiply by
Pennyweights (dwt)	Ounces (Troy)	0.05
Pennyweights (dwt)	Grams	1.555174
Pennyweights (dwt)	Grains	24.00
Ounces (Troy)	Pennyweights	20.001
Ounces (Troy)	Grams	31.103486
Ounces (Troy)	Grains	480
Grains	Ounces (Troy)	0.002083
Grains	Grams	0.064789
Grains	Pennyweights	0.041666
Grams	Pennyweights	0.6430
Grams	Grains	15.4324
Grams	Ounces	0.03215
Cu. Yard	Cu. Metres	0.76455
cu. metres	Cu. Yards	1.30795

A unit of weight for precious stones is equivalent to 200 milligrams (1/5 g) or 3.08647 $\frac{1}{8}$.

STRATEGIC IMPORTANCE

A good fraction of gold produced is held in central banks where it is used as a reserve stock and to settle international trade balances.

Being the most malleable and ductile but soft metal, it is usually alloyed to give strength. Gold is a good conductor of heat and electricity and it is unaffected by most reagents. It is used as a standard for monetary systems in many countries. It belongs to groups of precious metals that have found widespread uses mainly as alloys with platinium group of metals, jewelleries, decorative arts and other ornamental purposes. Other uses include manufacturing of spinnets, in making synthetic fibres, thermocouples and in electronic industries. Gold is also used as protective and decorative coating for other metals in architectural designs as well as coatings in aircraft engines and earth satelites. It is a good reflector of infrared and is inert. ^{198}Au with a half life of 2.7 is used for treating cancer and other diseases.

Purple of cassius (gold in purple) is used in manufacturing red and purple glass and in colouring enamels and painting porcelain. Many gold-nickel and gold-iron alloys are magnetic are used in computer memory devices. Gold alloyed with palladium and copper are used in dental work, and gold alloyed with only palladium is used for the production of fusion resistance laboratory ware.

A good proportion of gold produced is held in personal reserves. Considering the uses the people have made of gold for centuries, it is not a surprise that it is called the king of metals.

APPENDIX 4

STOCHIOMETRIC COMPUTATIONS FOR SODIUM CYANIDE CONCENTRATIONS

(I) Gold cyanidation reaction according to Habashi(1966, 1967) is given as:

$2Au + 4NaCN + O_2 + 2H_2O \rightarrow 2NaAu(CN)_2 + 2NaOH + 2H_20_2$

That is,

$$\begin{aligned} 2 \text{ parts of Au} &\equiv 4 \text{ moles of NaCN} \\ \Rightarrow\ 2(197.967) &\equiv 4(23+12+14) \\ \Rightarrow\ 395.934 \text{ g Au} &\equiv 196 \text{ g NaCN} \\ \Rightarrow\ 1 \text{ g Au} &\equiv \frac{196}{395.934} \text{ g NaCN} \\ &\equiv 0.4950 \text{ NaCN} \end{aligned}$$

(II) Analytical Au for the unsized concentrate

$$\begin{aligned} \text{Weight of sample} &= 12.5050\text{g} \\ \text{ppm of Au in Sample} &= 4.666 \\ \%\text{Au} &= \frac{\text{ppm} \times \text{ volume} \times \text{dilution factor} \times 100}{\text{Sample weight} \times 10^6} \\ &= \frac{\text{ppm} \times \text{ volume} \times \text{dilution factor}}{\text{Sample weight} \times 10^4} \\ &= \frac{4.666 \times 50}{12.5050 \times 10^4} = 0.0019\%\ \text{Au} \\ \text{Gold content} &= \%\text{Au} \times \text{Weight} = \frac{0.0019}{100} \times 12.5050 = 0.0002389\text{g Au} \end{aligned}$$

$$\begin{aligned} \text{Recall from (I) that 1g of Au} &\equiv 0.4950 \text{ NaCN} \\ \Rightarrow\ 0.000238\text{g Au} &= 0.4950 \times 0.000238\text{g CN} \\ &= 0.0001178\text{g of CN} \end{aligned}$$

(III)

$$\% \text{ CN} = \frac{\text{mass of cyanide} \times 100}{\text{volume}}$$

$$\Rightarrow \text{volume} = \frac{\text{mass of cyanide} \times 100}{\%\text{CN}}$$

Thus for 0.05% CN,

$$\text{volume} = \frac{0.000178 \times 100}{0.05} = 0.2356 \text{ ml}$$

$$0.0001178\text{g CN} \equiv 0.2356\text{ml}$$

$$\Rightarrow 1g \text{ CN} = \frac{0.2356}{0.0001178} = 2000\text{ml of water}$$

For 0.05% CN, $1\text{g} \equiv 2000\text{ml}$

$\Rightarrow$ 0.1% CN, $1\text{g} \equiv \frac{2000}{2} = 1000\text{ml}$

0.2% CN, $1\text{g} \equiv \frac{2000}{4} = 500\text{ml}$

0.3% CN, $1\text{g} \equiv \frac{2000}{6} = 333.33\text{ml}$

APPENDIX 5

LABORATORY REPORT

REPUBLIC OF ZAMBIA

MINISTRY OF MINES AND MINERALS DEVELOPMENT

GEOLOGICAL SURVEY DEPARTMENT

P.O. Box 50135, Lusaka
Telephone: 251655
Telegrams: Geocentral

LABORATORY REPORT

To: Mr. John Ade
School of Mines
191

Sample received 14/03/95
Lab. Ref. No. [illegible]
Date reported [illegible]

Nature of Sample: Nigeria

Source of Sample:

Examination Required: Fire - Assay for Gold

REPUBLIC OF ZAMBIA
DISPATCH
19 MAR 1996
GEOLOGICAL SURVEY DEPARTMENT
P.O. BOX 50135, LUSAKA

RESULTS OF INVESTIGATION

The results of gravimetric Fire - Assay tests on the samples were as follows

SAMPLE NUMBER	SIZE FRACTION (MICRONS)	G/T Au
1	+ 2,000	9.2 (7)
2	- 2,000 + 850	12.4 (5)
3	- 850 + 600	9.6 (6)
4	- 600 + 300	20.0 (4)
5	- 300 + 150	25.0 (3)
6	- 150 + 75	26.2 (2)
7	- 75	[illegible] (1)

Andrew Banda
Senior Metallurgist

APPENDIX 6

CALIBERATION TABLES FOR AAS

Table 4.12: Caliberation Table for AAS Analysis for Gold

Standards (ppm)	Absorbance $\times 10^{-3}$	Average Absorbance
Blanks	0,0	0
2.5	11,12	0.0115
5.0	26,26	0.026
7.5	40,36	0.038
10.0	53,50	0.0515
12.5	61,60	0.0605

Table 4.13: Caliberation Table for AAS Analysis for Silver

Standard Samples (ppm)	Absorbance $\times 10^{-3}$	Average Absorbance	Sensitivity's S=0.0044×2 /Absorbance
Blank	0,0		
2	52,51	0.051	0.173
4	100,95	0.047	0.187
6	134,128	0.043	0.205
8	203,194	0.045	0.196
12	320,316	0.0044	0.200

Table 4.14: Caliberation Table for AAS Analysis for Copper

Standard Samples (ppm)	Absorbance $\times 10^{-3}$	Average Absorbance	Sensitivity's S=0.0044×2 /Absorbance
Blank	0,0		
2.5	47,46	0.047	0.234
5.0	100,108	0.046	0.239
7.5	156,152	0.048	0.229
10.0	207,208	0.044	0.234
12.5	275,272	0.047	0.250

Table 4.15: Caliberation Table for AAS Analysis for Lead

Standard Sampies (ppm)	Absorbance $\times 10^{-3}$	Average Absorbance	Sensitivity's S=0.0044×2 /Absorbance
Blank	0,0		
5	10,14	0.010	2.200
10	35,39	0.014	1.571
15	61,68	0.010	2.000
20	106,104	0.015	1.467
25	153,150	0.012	1.833

Table 4.16: Caliberation Table for AAS Analysis for Zinc

Standard Samples (ppm)	Absorbance $\times 10^{-3}$	Sensitivity
Blank	0,0	0
0.2	14,12	0.013
0.4	32,34	0.033
0.6	47,49	0.048
0.8	68,65	0.0665
1.0	101,102	0.1015

APPENDIX 7

PERCENTAGE GOLD DETERMINATION, GOLD CONTENT IN GRAMMES, AAS DETECTION LEVEL AND AAS SENSITIVITY

(i) $$\%Au = \frac{ppm \times volume \times dilution\ factor \times 100}{Sample\ weight \times 10^6}$$

$$= \frac{ppm \times volume \times dilution\ factor}{Sample\ weight \times 10^4}$$

(ii) Gold content in grammes = % Au × weight of sample used.

(iii) Detection Level = 2 × Standard deviation.

(iv) Sensitivity, $$s = \frac{0.0044 \times lowest\ standard}{absorbance\ for\ the\ lowest\ standard}$$

APPENDIX 8

TABLES ON EXPERIMENTATION

Table 4.17: Effect of Cyanide Concentration on Gold Recovery.

Sodium Cyanide Concentration	Absorbance $\times 10^{-3}$			Average Absorbance	Gold Recovery (ppm)
	1	2	3		
0.05%	5	4	4	0.0043	0.0265
0.1%	9	8	7	0.008	0.1176
0.2%	24	27	23	0.0247	0.1776
0.3%	42	40	36	0.039	1.4429

pH = 11.5

Detection Time = 6 hours

Sample: Unsized concentrate

Table 4.18: Effect of pH on Gold Recovery

pH	Absorbance $\times 10^{-3}$		Average Absorbance	Gold Recovery (ppm)
	1	2		
11.5	129	131	0.130	4.4865
12	87	87	0.087	4.4112
12.5	35	38	0.0365	1.9739
13	81	79	0.080	4.0734

Cyanide concentration = 0.3%

Retention Time = 6 hours

Sample: Unsized Concentrate

Table 4.19: Effect of Particle Size on Gold Recovery

Particle Size μm	Absorbance $\times 10^{-3}$ 1	2	Average Absorbance	Gold Recovery (ppm)
+2000	-	-	-	-
-2000 +850	14	15	0.0145	0.6375
-850 +600	118	114	0.116	4.4879
-600 +300	30	31	0.0305	1.6336
-300 +150	51	49	0.050	2.7097
-150 +75	195	199	0.1975	10.8499
-75	178	176	0.177×10	97.185

Cyanide concentration = 0.3%

Retention time = 6 hours

pH = 11.5

Samples: 6 particle size fractions as shown in the table

APPENDIX 9

FLOWSHEET DEVELOPMENT BASED ON HEAP LEACHING

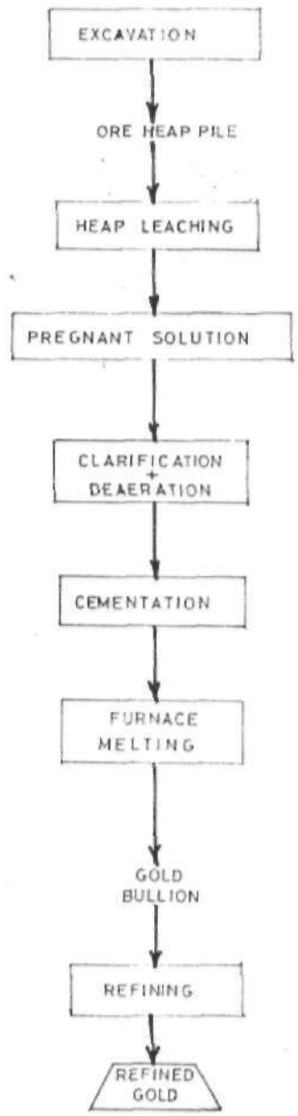


Fig.4.13 Flowsheet based on Heap leaching